WL-TR-94-XXX
NMERI 1994/40/31790

THE DEVELOPMENT OF CF_3I AS A HALON REPLACEMENT

Ted A. Moore, Stephanie R. Skaggs, Min R. Corbitt, and
Robert E. Tapscott

NEW MEXICO ENGINEERING RESEARCH INSTITUTE
The University of New Mexico
Albuquerque, New Mexico 87131-1376

and

Douglas S. Dierdorf

PACIFIC SCIENTIFIC
HTL/Kin Tech Division
3916 Juan Tabo, NE, Suite 12
Albuquerque, NM 87111

and

Charles J. Kibert

WRIGHT LABORATORIES (WL/FIVCF)
139 Barnes Drive, Suite 2
Infrastructure Technology Section
Tyndall Air Force Base, Fl 32403-5319

November 1994

Final Report for Period September 1993 through November 1994

Prepared for

WRIGHT LABORATORIES (WL/FIVCF)
139 Barnes Drive, Suite 2
Infrastructure Technology Section
Tyndall Air Force Base, Florida 32403-5319

NOTICE

This report has been reviewed by the Public Affairs (PA) Officer and is releasable to the National Technical Information Service (NTIS). At NTIS it will be available to the general public, including foreign nationals.

CHARLES J. KIBERT
Project Officer

EDGAR ALEXANDER
Chief, Air Base Technology Branch

RICHARD N. VICKERS
Chief, Infrastructure Technology Section

Please do not request copies of this report from WL/FIVC. Additional copies may be purchased from:

NATIONAL TECHNICAL INFORMATION SERVICE
5285 PORT ROYAL ROAD
SPRINGFIELD, VIRGINIA 22161

Federal government agencies and their contractors registered with Defense Technical Information Center should direct requests for copies of this report to:

DEFENSE TECHNICAL INFORMATION CENTER
CAMERON STATION
ALEXANDRIA, VIRGINIA 22314

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 074-0188

1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE
October 1994

3. REPORT TYPE AND DATES COVERED
Final report, Sept. 1993-Nov. 1994

4. TITLE AND SUBTITLE
The Development of CF_3I as a Halon Replacement

5. FUNDING NUMBERS
S-5000.7

6. AUTHOR(S)
Ted A. Moore, Stephanie R. Skaggs, Min R. Corbitt, Robert E. Tapscott, Douglas S. Dierdorf and Charles J. Kibert

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

New Mexico Engineering Research Institute
The University of New Mexico,
Albuquerque, New Mexico 87131-1376

Pacific Scientific HTL/Kin Tech Division
3916 Juan Tabo, NE, Suite 12
Albuquerque, NM 87111

Wright Laboratories (WL/FIVCF)
139 Barnes Drive, Suite 2
Infrastructure Technology Section
Tyndall Air Force Base, FL 32403-5319

8. PERFORMING ORGANIZATION REPORT NUMBER
NMERI 1994/40/31790

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)

Engineering & Services Program Office
Air Force Civil Engineering Support Agency
Tyndall AFB, FL 32403-6001

SERDP
901 North Stuart St. Suite 303
Arlington, VA 22203

10. SPONSORING / MONITORING AGENCY REPORT NUMBER
WL-TR-94-XXX

11. SUPPLEMENTARY NOTES
Prepared for Wright Laboratories (WL/FIVCF). This work was supported in part by S-5000.7.

12a. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release: distribution is unlimited

12b. DISTRIBUTION CODE
A

13. ABSTRACT ***(Maximum 200 Words)***
This report summarizes the testing efforts on CF_3I as a halon replacement agent; these efforts were supported by the CF_3I Ad Hoc Working Group. The Working Group included representatives from the US Air Force, Army, Navy, North Slope oil and gas producers, Pacific Scientific, and West Florida Ordnance. The testing efforts confirmed the laboratory-scale fire suppression and explosion inertion data, determined key acute toxicological information, assessed global environmental characteristics, identified manufacturing sources for developmental quantities of agent, and determined agent stability and materials compatibility properties. The n-heptane cup-burner concentration was 3.1 percent and the propane inertion concentration at peak flammability was 6.2 percent. The atmospheric lifetime is less than 1 day, the ODP is less than 0.008 and more likely 0.0001, and the 20-year GWP is less than 5 relative to CO_2. Toxicological tests showed that CF_3I has a low order of acute toxicity with the 15-minute rat LC_{50} equal to 27.4 percent. The dog cardiac sensitization NOAEL was 0.2 percent and the LOAEL was 0.4 percent. Since the expected design concentration will be in the range of 5 to 7 percent, the cardiotoxicity profile will preclude the use of CF_3I for total-flooding applications in normally occupied areas. However, CF_3I is still a highly promising replacement for unoccupied areas and streaming applications.

14. SUBJECT TERMS
CF_3I, halon replacement, streaming agent, total-flooding agent, inerting agent, SERDP

15. NUMBER OF PAGES
110

16. PRICE CODE
N/A

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
unclass	unclass	unclass	UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

PREFACE

This report was prepared by the Advanced Protection Technologies (APT) Division, New Mexico Engineering Research Institute (NMERI), The University of New Mexico, Albuquerque, New Mexico, 87131-1376 for the Infrastructure Technology Section of Wright Laboratories (WL/FIVCF), Tyndall Air Force Base, Florida 32403-5319, under Contract S-5000.7, Project No. 31790 from Applied Research Associates (ARA).

The program Start Date was 27 September 93, and the End Date was 31 December 94. The WL/FIVCF Project Officer was Charles J. Kibert. The NMERI Principal Investigator was Stephanie R. Skaggs.

The support of the US Air Force, US Army, US Navy, US Environmental Protection Agency (EPA), North Slope Oil and Gas Producers, Pacific Scientific Company, West Florida Ordnance, and the other members of the CF_3I Ad Hoc Working Group for the development of the information presented in this document is acknowledged.

NMERI 1994/40/31790

(The reverse of this page is blank.)

EXECUTIVE SUMMARY

A. OBJECTIVE

The objective of this report is to provide documentation on the developments to date on trifluoroiodomethane (CF_3I) as a halon replacement. Results to date are summarized and future requirements are outlined. This work has been sponsored by the CF_3I Ad Hoc Working Group, which includes representatives from the US Air Force (USAF), US Army, US Navy, North Slope Oil and Gas Producers, Pacific Scientific Company, and West Florida Ordnance.

B. BACKGROUND

Halon production ceased at the end of 1993, and, as yet, the search for an ideal halon replacement has not been successful. A large number of candidate replacement agents have been announced by industry for commercialization, and additional chemicals are under consideration. Most of the announced agents are so-called "first-generation" agents—hydrochlorofluorocarbons (HCFC), hydrofluorocarbons (HFC), perfluorocarbons (PC or PFC), or hydrobromofluorocarbons (HBFC). Each of the first-generation agents has significant drawbacks when considered in terms of the "idealized halon replacement." Some have non-zero ozone-depletion potentials (ODP). Others have zero ODPs, but have relatively long atmospheric lifetimes and high global warming potentials (GWP). From an effectiveness standpoint, 1.5 to 3.0 times more agent is required, compared with Halon 1211 and Halon 1301.

"Second-generation" agents are designed specifically to avoid these tradeoffs. The most promising second-generation agent identified to date is CF_3I. To expedite development of CF_3I, the direct involvement of the user community has been required in order to move toward commercialization. No single company has been willing to accept all the development costs and risks associated with bringing CF_3I to market.

When CF_3I was announced as a potential halon replacement agent, little to no information, other than its high fire extinguishing effectiveness and suspected low atmospheric lifetime, was available. Only laboratory quantities were available (25 to 1000 grams) at roughly \$0.95/gram (\$430/pound). Minimal toxicity information was known. The actions documented herein have made CF_3I a commercial product in less than 18 months (CF_3I is now available in 1000-pound quantities at \$50-75/pound).

C. SCOPE

On May 13, 1993, a group of interested users met during the 1993 Halon Alternatives Technical Working Meeting in Albuquerque, New Mexico, to decide whether and how to pursue the positive lead represented by CF_3I. Initial funding members of the group were the USAF, US Army, US Navy, the North Slope Oil and Gas Producers, and Pacific Scientific Company. This group developed a series of five tasks aimed at identifying potential "show stoppers," which would prevent the ultimate development of CF_3I as a halon replacement. The five key tasks included the following:

(1) Confirm laboratory-scale fire extinguishing data.
(2) Determine key toxicological information.
(3) Determine the global environmental characteristics.
(4) Determine the manufacturability and source for developmental quantities of agent.
(5) Determine the stability and materials compatibility characteristics of CF_3I.

Follow-up meetings occurred throughout 1993 and 1994 to present the work of the New Mexico Engineering Research Institute (NMERI), National Institute of Standards and Technology (NIST), USAF, US Army, US Navy, Naval Research Laboratory (NRL), and others who were evaluating CF_3I.

D. RESULTS

Several physical and chemical properties of CF_3I have been determined. It is a dense liquid (liquid density = 2.10 g/mL) with a boiling point (-22.4 °C) somewhat higher than Halon 1301. Based upon confirmation of previous laboratory data (cup-burner value = 3.1 percent, propane flammability peak = 6.5 percent) and results of small and intermediate-scale testing to date, CF_3I is considered to be an isovolumetric halon replacement for Halon 1301 in most applications. The recommended fire suppression and propane inertion design concentrations are 5.0 and 7.0 percent, respectively, the same as for Halon 1301. CF_3I has also been shown to be a "drop-in" replacement for Halon 1211 in streaming applications.

The amount of toxicological information on CF_3I has increased dramatically in a relatively short time span. Within a year, acute inhalation studies, cardiac sensitization tests, and preliminary genetic toxicity tests were performed. Acute inhalation studies revealed that CF_3I has a low order of toxicity with a 15-minute rat LC_{50} equal to 27.4 percent. Effects during acute studies included anesthesia, salivation, and audible respiration. Serum chemistry measurements showed no abnormal results. Cardiac sensitization measurements indicated that the no observable adverse effect level (NOAEL) is 0.2 percent and the lowest observable adverse effect level (LOAEL) is 0.4 percent. Since the expected design concentration will be in the range of 5.0 to 7.0 percent, this cardiotoxicity profile precludes the use of CF_3I for total-flooding applications in normally occupied areas. However, CF_3I is still a highly promising alternative in total flooding of unoccupied areas and for streaming applications. The potential for carcinogenic effects has not yet been completely evaluated. Although the screening test results are mixed, the available information is insufficient to conclude that CF_3I is unsafe in firefighting applications. Further testing (90-day subchronic test) will need to be performed to evaluate the preliminary carcinogenic potential in whole animal systems.

CF_3I is readily photolytized in the near ultraviolet (uv) light spectrum, resulting in an atmospheric lifetime of less than 1 day. The latest estimated ODP for CF_3I is less than 0.008 and more likely below 0.0001. Due to the short atmospheric lifetime of the chemical and the photolytic decomposition mechanism, the 20-year GWP based upon CO_2 will be less than 5.

There are two declared US manufacturers of CF_3I and other fluoroiodocarbons (FIC): Pacific Scientific and West Florida Ordnance (WFO). Pacific Scientific is in the process of increasing production from 50 to 500 pounds/day and has built a new plant in Oklahoma that will have an ultimate 3-5 million pounds/year capacity. WFO has a capacity of 100 pounds/8-hour shift and, depending on demand, could operate multiple production shifts. WFO is in the process of constructing a new plant in Tennessee. There are also several US producers of laboratory quantities of FIC chemicals, as well as production in locations such as Russia and Japan. Long-term price predictions for large quantity purchases of CF_3I vary widely, from a low of $10 to about $25/pound for annual production quantities of several million pounds.

A Significant New Alternatives Policy (SNAP) application was submitted to the US Environmental Protection Agency (EPA) on behalf of Pacific Scientific for CF_3I under the tradename Triodide® in May 1994 for total-flood application in unoccupied spaces. Triodide® was proposed to be acceptable for unoccupied areas in August 1994. In September 1994, a SNAP application was submitted on behalf of Pacific Scientific for CF_3I for the use of Triodide® in streaming applications. The streaming application submittal is awaiting EPA action.

As with Halon 1301, the chemical appears to be compatible with normal construction materials (stainless steel, carbon steel, brass, and aluminum) to temperatures of at least 77 °C (170 °F). The chemical also appears to be compatible with ethylene-propylene diene monomer

(EPDM), nitrile, and neoprene rubbers to 77 °C (170 °F). The chemical appears to be stable indefinitely in the absence of light, oxygen, and water at temperatures below 116 °C (240 °F).

E. CONCLUSIONS

The toxicological, environmental, and firefighting results to date indicate that CF_3I is a highly promising halon replacement candidate for unoccupied areas and streaming applications. The testing of CF_3I was initiated 18 months ago and has thus far demonstrated that CF_3I meets the criteria established for an environmentally acceptable, high efficiency replacement for Halon 1301 in unoccupied areas and Halon 1211 for certain applications. CF_3I is commercially available in bulk. This rapid development is the result of a concerted effort by a number of personnel and organizations from several disciplines with the foresight to obtain an agent that more completely meets the needs of the halon user community. In unoccupied applications CF_3I is an excellent replacement. Note, however, risk assessments for different types of firefighting applications and for handling the chemical during manufacture, normal maintenance, storage, and accidental discharge are required prior to using CF_3I as a fire suppression or inertion agent.

F. RECOMMENDATIONS

The following recommendations are made:

(1) Large-scale testing should be performed based upon user-specific applications to confirm the effectiveness results developed to date. Large-scale testing would include that required for UL and FM listings.
(2) Studies of decomposition products resulting from the extinguishing process should be performed.
(3) Nitrogen solubility should be determined for CF_3I.
(4) The thermal operating envelope for CF_3I should be developed.
(5) The hot surface (pyrolysis) products of CF_3I should be determined.
(6) The subchronic and developmental toxicity should be determined. Additional toxicity studies may be required if adverse effects for endpoints are determined in the subchronic test.
(7) CF_3I blends should be investigated to determine whether toxicity considerations could be reduced and extinguishment efficiency maintained.

TABLE OF CONTENTS

TABLE OF CONTENTS (CONTINUED)

TABLE OF CONTENTS (CONCLUDED)

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS AND ACRONYMS

APG	Aberdeen Proving Ground
APU	aircraft power unit
ASTM	American Society for Testing and Materials
Buna N	nitrile-butadiene rubber
CAA	chemical action agent
CAS	Chemical Abstracts Service
CFC	chlorofluorocarbon
ECG	electrocardiographic
EPA	Environmental Protection Agency
EPDM	ethylene-propylene diene monomer
FIC	Fluoroiodocarbon
FTIR	fast Fourier transform infared
GWP	global warming potential
HFC	hydrofluorocarbon
HCFC	hydrochlorofluorocarbon
HBFC	hydrobromofluorocarbon
FM	Factory Mutual, Inc.
LC_{50}	lethal concentration to kill 50 percent of a population
LLNL	Lawrence Livermore National Laboratories
LOAEL	lowest observable adverse effect level
NBR	nitrile-butadiene rubber
NIST	National Institute of Standards and Technology
NFPA	National Fire Protection Association
NMERI	New Mexico Engineering Research Institute
NOAEL	no observable adverse effect level
NRL	Naval Research Laboratory
ODP	ozone-depletion potential
PAA	physical action agent
QSAR	quantitive structural activity relationship
SNAP	Significant New Alternatives Policy
SVEq	Storage Volume Equivalency
UL	Underwriters Laboratories
USAF	United States Air Force
US	United States
VEq	Volume Equivalency
WEq	Weight Equivalency
WFO	West Florida Ordnance
WL	Wright Laboratories

SECTION I
INTRODUCTION

A. BACKGROUND

Halon production ceased at the end of 1993, and, as yet, the search for an ideal halon replacement has not been successful. A large number of candidate replacement agents have been announced by industry for commercialization, and additional chemicals are under consideration. Most of the announced agents are so-called "first-generation" agents, hydrochlorofluorocarbons (HCFC), hydrofluorocarbons (HFC), perfluorocarbons (PC or PFC), or hydrobromofluorocarbons (HBFC). Each of the first-generation agents has significant drawbacks when considered in terms of the "idealized halon replacement." Some have non-zero ozone-depletion potentials (ODP). Others have zero ODPs, but have relatively long atmospheric lifetimes and high global warming potentials (GWP). From an effectiveness standpoint, 1.5 to 3.0 times more agent is required, compared with Halon 1211 and Halon 1301.

"Second-generation" agents are designed specifically to avoid these tradeoffs and have required the direct involvement of the user community to move toward commercialization.

1. Ideal Halon Replacement (Figure 1)

Successful halon replacement agents have a minimum of four requirements:

(1) Low global environmental impact (i.e., low ozone-depletion potential [ODP], global warming potential [GWP], and atmospheric lifetime)
(2) Acceptable toxicity to suit the application
(3) Low residue (i.e., clean and volatile)
(4) Effectiveness against fires and/or explosions

Although it is relatively easy to find replacements that fulfill any three of these requirements, no "first-generation" replacement has been found that meets all four. The most difficult requirement appears to be finding replacements that have acceptable global environmental impact, an effectiveness equal to the present halons, and low toxicity. Any replacement must also be manufacturable at an affordable cost.

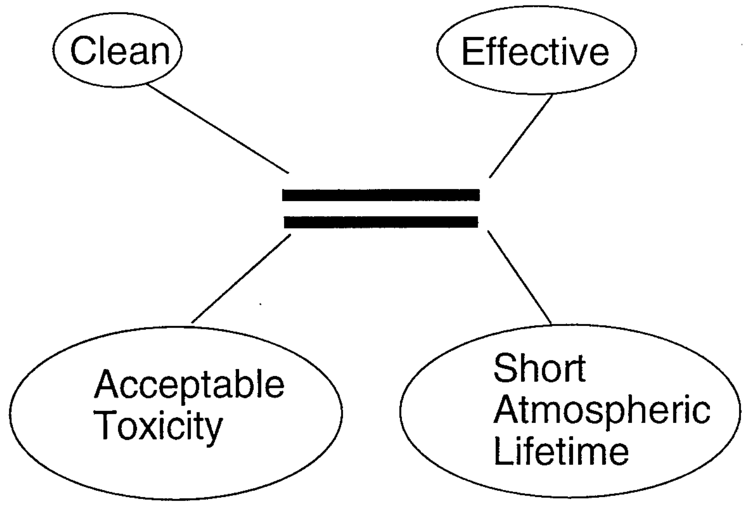


Figure 1. Ideal Halon Replacement.

2. Definitions

Halon Substitutes --- A potential agent that is chemically similar to the present halons (e.g., a halocarbon) is referred to as a halon "replacement"; "alternatives" are those agents that are "not-in-kind" substitutes because their chemical nature is different from halons. Alternatives include carbon dioxide, foam, water, dry chemicals, and inert gases. Both replacements and alternatives are referred to as halon substitutes.

Physical Action Agents (PAA) --- These chemicals act primarily by cooling and dilution. PAAs are generally less effective than CAAs.

Chemical Action Agents (CAA) --- These chemicals act primarily by inhibiting the chain reactions that maintain combustion.

First-Generation Agents --- First-generation agents are halocarbons from families that have been commercially available for several years. Four chemical families comprise first-generation replacements: hydrobromofluorocarbons (HBFC), hydrochlorofluorocarbons (HCFC), hydrofluorocarbons (HFC), and perfluorocarbons (FC or PFC). Many compounds being considered as chlorofluorocarbon (CFC) replacements in refrigeration, air conditioning, foam blowing, and solvents are found in the HCFC, HFC, and FC families.

The HBFCs—like the halons—are CAAs and are very effective extinguishing agents. However, production of HBFC materials is scheduled to end December 31, 1995. The other chemical families are generally less effective fire extinguishants than the existing halons (requiring 1.5 - 3.0 times more agent and system changes). Table 1 is a summary of the key first-generation chemical families.

Second-Generation Agents --- Second-generation agents were either unavailable or available only at high cost (@ \$100/kg) and in small quantities during May 1993 time frame.

TABLE 1. FIRST-GENERATION HALON REPLACEMENT CHEMICAL FAMILIES WITH COMPARISON TO CF_3I

Family	Extinguishment Mechanism	Toxicity	ODP	Atmospheric Lifetime	Extinguishment Effectiveness	Montreal Protocol Restrictions
HBFC	CAA	Moderate	High	Low	Excellent	Phaseout 1996
HCFC	PAA	Low	Low	Low	Fair to Good	Phaseout 2010-2030
HFC	PAA	Low	Zero	Moderate	Fair	No Restrictions
FC	PAA	Very Low	Zero	High	Fair	No Restrictions
[a]CF_3I	CAA	Moderate to High	Essentially Zero	Very Low	Excellent	No Restrictions

[a]For comparison.

3. Drawbacks of First-Generation Agents

Several first-generation total-flood agents were addressed in the recently published National Fire Protection Association (NFPA) 2001 Standard (Reference 1) and the EPA Significant New Alternatives Policy (SNAP) Program list (Reference 2). Each of these agents has significant drawbacks when considered in terms of the "ideal halon replacement" described previously. The HBFCs and HCFCs have non-zero ODPs, with HBFCs having the highest. HFCs and FCs have zero ODPs, but can have relatively long atmospheric lifetimes and high GWPs, particularly in the case of the FCs. The PAAs often form decomposition products during fire extinguishment (References 3 and 4). The FCs and HFCs are under regulatory pressure due to their long atmospheric lifetimes and high GWP. Therefore, there is a need to develop an agent with suitable environmental, toxicological, and firefighting attributes. As a consequence, second-generation agent development was initiated. Key properties and drawbacks are described in Table 2.

B. SECOND-GENERATION AGENTS

As discussed above, the primary distinguishing characteristics of second-generation agents is the lack of economical large-scale manufacturing capacity and limited toxicity information. The primary problem in evaluating an agent is lack of chemical available at reasonable cost to support testing (effectiveness and toxicity).

1. Generic Classifications and Mechanisms for Elimination from the Atmosphere

Earlier work (Reference 5) provided key insight into the specific characteristics necessary for near zero or zero ODP agents. This work lead to a detailed description of key second generation agent families (Reference 6). Table 3 lists the key families of second-generation agents and the properties projected to prevent negative global environmental impacts with superior fire suppressant and explosion prevention effectiveness.

2. Selection of Fluoroiodocarbons for Accelerated Development

Several key factors were important in selecting the FICs over the other families for immediate development:

(1) Literature data indicated that the toxicity could be acceptable.

(2) Known fire extinguishing characteristics were exceptional.

(3) Simple methods for preparation suggested that commercial scale-up would be practical (Reference 7).

The combination of these factors created a demand among members of the halon user community to advance the development of CF_3I as a possible halon replacement. A working group was, therefore, formed.

C. CF_3I WORKING GROUP

On May 13, 1993 a group of interested users met during the 1993 Halon Alternatives Technical Working Meeting in Albuquerque, NM, to decide how to pursue the positive lead represented by CF_3I. Key initial sponsoring members of the group were representatives of the

TABLE 2. FIRE SUPPRESSION ANALYSIS AND DRAWBACKS OF COMMERCIALIZED AGENTS WITH COMPARISON TO HALON 1301.

Agent[a]	Recommended Design Conc.[a]	WEq[b]	VEq[b]	Drawbacks
FC-3-1-10 (CEA-410)	6[c]	1.9	1.7	Long atmospheric lifetime, high GWP
HBFC-22B1 (FM 100)	4.9	0.9	1.0	High toxicity, Montreal Protocol phase out Jan. 1, 1996, high ODP
HCFC Blend A (NAF SIII)	8.6[c]	1.1	1.4	Begin phaseout 2010
HCFC-124 (FE 241)	8.5	1.6	1.6	Begin phaseout 2010
HFC-125 (FE 25)	10.9	1.9	2.3	Cardiotoxic at design concentration, long atmospheric lifetime, high GWP
HFC-227ea (FM 200)	7	1.7	1.6	Moderate GWP, marginally cardiotoxic at design concentration, unacceptable for occupied inertion applications due to cardiotoxicity
HFC-23 (FE 23)	16	1.7	2.2	Long atmospheric lifetime, high GWP, requires high pressure system
IG-541 (INERGEN)	37.5	2.0	10.5	Low effectiveness, toxicity questions, high weight and volume requirement
CF_3I (Triodide®, Iodoguard®)	5.0[d]	1.4	1.0	Cardiotoxic at design concentration
Halon 1301	5.0[e]	1.0	1.0	

[a]Recommended in NFPA 2001 (1994) as 120 percent of cup-burner value for *n*-heptane.

[b]Relative to Halon 1301 = 1.0.

[c]Based on listing/approval tests.

[d]Manufacturers recommended design concentration (170 percent of cup-burner value).

[f]For comparison

[e]Recommended design concentration in NFPA 12A (170 percent of cup-burner value).

TABLE 3. KEY FAMILIES OF SECOND-GENERATION AGENTS.

Family	Example	Mechanism of Atmospheric Elimination
Fluoroiodocarbons	CF_3I, C_3F_7I	Iodine also helps reduce the tropospheric lifetime through photolysis, which reduces the ODP and GWP.
Bromofluoroalkenes	$CF_2BrCF_2CH{=}CH_2$	The double bond leads to a short atmospheric lifetime due to rapid reaction with hydroxyl free radicals in the troposphere.
Bromofluoroethers	$CHBrFCF_2\text{-}O\text{-}CH_3$	The oxygen helps reduce the atmospheric lifetime through rainout, thus lowering the ODP and GWP.

US Air Force, US Army, and US Navy; the North Slope Oil and Gas Producers; and Pacific Scientific. This group developed the following five tasks aimed at identifying potential "show stoppers," which would prevent the ultimate development of CF_3I.

(1) Confirm laboratory scale fire extinguishing data.
(2) Determine key toxicological information.
(3) Determine the global environmental characteristics.
(4) Determine the manufacturability and source for development quantities of agent.
(5) Determine the stability and materials compatibility characteristics of CF_3I.

Follow-up meetings occurred throughout 1993 and 1994 to present the work of NMERI, NIST, US Air Force, US Army, US Navy, and others in evaluating CF_3I. The results of this community-wide effort are detailed in the following sections of this report. The following sections summarize the status of CF_3I development as of October 1994. A list of active meeting participants is included in Appendix A. Appendix B is a compilation of the newsbriefs sent to members of the working group.

SECTION II
PHYSICAL PROPERTIES

CF_3I is a dense gas with a boiling point somewhat higher than Halon 1301. Table 4 includes a listing of the available key physical and chemical properties of CF_3I. The higher boiling point may restrict its use in some applications, such as military aircraft fuel tank inertion where the chemical must remain gaseous even at very cold temperatures. The boiling point may also provide advantages in other uses where a higher boiling point might prove useful, such as in steaming applications.

TABLE 4. PHYSICAL AND CHEMICAL PROPERTIES OF CF_3I.

Physical or Chemical Property	Value or Property
Chemical Abstracts Service (CAS) No.	2314-97-8
Molecular Weight	195.91
Physical State @ 25 °C	Gas
Melting Point	Unknown
Boiling Point @ 1 atm. pressure	-22.5 °C (-8.5 °F)
Liquid Density @ -32.5 °C	2.36 g/mL
Liquid Density @ 25 °C	2.096 g/mL
Odor Threshold	Odorless
Vapor Pressure	78.4 psia @ 25 °C (78 °F)
Pressure Temperature Curve	log P (psia) = 5.7411-1146.82/T(K)
Critical Pressure	586 psia (est.)
Critical Temperature	122 °C (est.) (252 °F)
Critical Volume	225 cm^3/mol (est.)
Heat of Formation	-141 kcal/mol
Heat of Vaporization	5.26 kcal/mol
Electron Affinity	150 ± 20 kJ/mol
Refractive Index (Liquid) @ -42 °C	1.379
Dipole Moment	1.68 debye (calculated)
Vapor Heat Capacity	16.9 cal/mol-K
C-I Bond Dissociation Energy	54 kcal/mol

An American Society for Testing and Materials (ASTM) draft standard has been developed concerning purity requirements for CF_3I (Appendix C) to ensure the chemical and physical integrity of CF_3I.

SECTION III
FIRE SUPPRESSION CHARACTERISTICS

A. BACKGROUND

CF_3I was first evaluated as a fire suppression agent in 1947 by researchers at Purdue University during a study of halogenated compounds sponsored by the US Army (Reference 8). This study formed the basis for the development of halons as fire suppression agents. CF_3I was reevaluated as a halon replacement in the early 1990s by NMERI (Reference 9) and the NRL (Reference 10). Early indications were that CF_3I would behave almost identically to Halons 1301 and 1211 in fire suppression.

B. CUP-BURNER EXTINGUISHING CONCENTRATIONS

Cup-burner concentrations using a variety of fuels have been determined for CF_3I (Table 5). The recommended design extinguishing concentration for CF_3I is 5.0 percent by volume (same as Halon 1301). The design concentration of 5.0 percent provides a 70 percent (1.70) safety factor above the cup-burner value (same as Halon 1301), providing minimum decomposition products. For new installations, a lower design concentration could be used.

C. INERTION CONCENTRATION TESTING

Figure 2 shows the flammability diagram for CF_3I and propane; Halon 1301 data are included for comparison. The inertion concentration at the flammability peak for propane is 6.5 percent (essentially the same as Halon 1301 at 6.2 percent). The test method description and results for other compounds can be found in Reference 11.*

* Moore, T.A., Skaggs, S.R., and Dierdorf, D.S., "Large-Scale Inertion Testing of NFPA 2001 Agents," presented at International Conference on CFC and Halon Alternatives, Washington, DC, 20-22 Oct 1993.

TABLE 5. NMERI CUP-BURNER VALUES FOR CF_3I AND HALON 1301 FOR A VARIETY OF FUELS.[a]

Fuel	Halon 1301[b]	CF_3I
Heptane	2.9	3.0
Propane	2.8	3.0
Methane	2.3	2.0
1-Butanol	3.7	3.3
Acetonitrile	1.5	1.7
AV Gas	2.8	3.7
Cyclopentanone	3.7	---
Diesel #2	2.6	3.3
Ethanol, Dehydrated Alcohol	3.0	3.0
Ethyl Acetate	1.9	3.0
Ethylene Glycol	1.9	2.4
Hydraulic Fluid #1	2.0	2.3
Turbo Hydraulic Oil 2380	2.2	2.1
JP-4	2.8	3.3
JP-5	2.6	3.2
Methanol	5.9	3.8
Methyl Ethyl Ketone	2.6	4.4
Methyl Isobutyl Ketone	2.4	2.9
Morpholine	3.9	---
n-Butyl Acetate	2.5	2.5
Pyrrolidine	2.9	2.8
Tetrahydrofuran	3.6	---
Unleaded Gasoline oxygenated with 7.8% ethanol	3.5	3.6
Xylene	1.7	5.5

[a]Test method description included in References 12 and 13.

[b]Recommended design concentration in NFPA 12A is 5.0 percent.

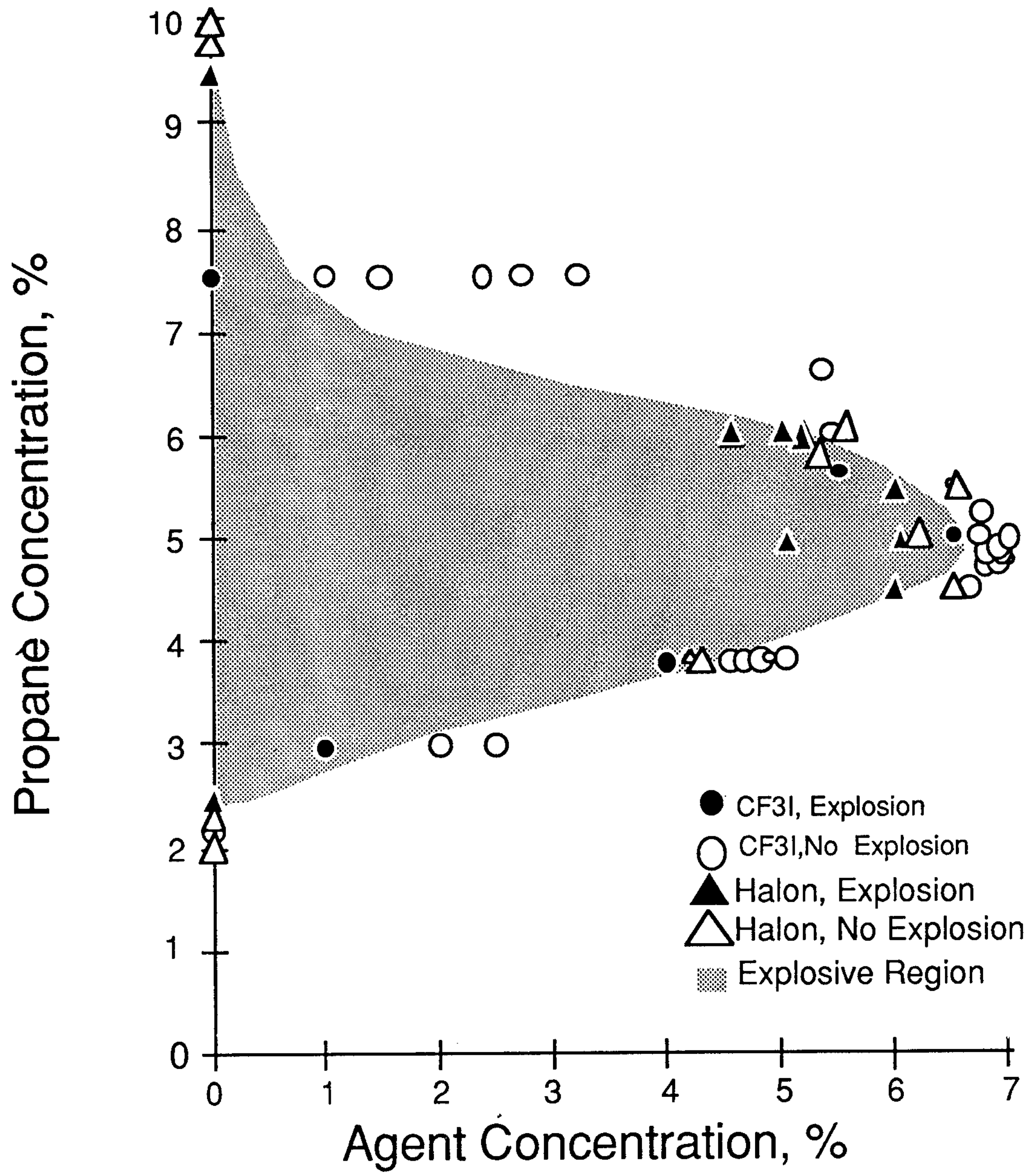


Figure 2. Flammability Diagram for CF_3I and Halon 1301.

D. INTERMEDIATE TO LARGE-SCALE TESTING

Additional extinguishment tests at Wright Laboratories (WL) (dry bay and engine nacelle)*; NRL (total flood) (Reference 14); Aberdeen Proving Ground (APG) (combat vehicle explosion suppression)†; and NMERI (combat vehicle engine compartment and total flood)‡ have shown performance near or equal to Halon 1301. Recent streaming agent testing at WL/FIVC Tyndall AFB, FL, has also shown that CF_3I performs better (on a pound-for-pound basis) than Halon 1211 in 20-pound portables on a 100-ft^2 JP-8 pool fire.

E. SUMMARY

Based upon laboratory and field-scale testing data to date, CF_3I is considered to be an isovolumetric halon replacement for Halon 1301 in most unoccupied applications. CF_3I has also been shown to be a replacement for Halon 1211 in streaming applications. Table 6 compares the replacement equivalency of CF_3I to Halon 1301. The same extinguishing quantity of CF_3I (5.0 percent design concentration) will weigh approximately 14 percent more but will occupy 8 percent less volume than Halon 1301 at 5.0 percent. Consequently, in space critical applications, CF_3I is an excellent potential replacement. Note, however, risk assessments related to design requirements (engineering considerations) and toxicity are required prior to using CF_3I as a fire suppression or inertion agent.

* Bennett, J. M., Personal communication, WL/FIVS, WPAFB, OH, March 10, 1994.

† Clauson, M., Personal communication, TACOM/TARDEC, Warren, MI, March 10, 1994, Albuquerque, NM.

‡ Moore, T. A., Personal communication, NMERI/UNM, Albuquerque, NM, March 10, 1994.

TABLE 6. REPLACEMENT EQUIVALENCY OF CF_3I COMPARED TO HALON 1301.

End Use	CF_3I Recommended Design Concentration, %	WEq	SVEq[a]
Fire Extinguishment	[b]5.0	1.36	1.00
Explosion Prevention (propane inertion)	[c]7.0	1.42	1.04

[a]Compared to Halon 1301 = 1.00.

[b]70 percent safety factor above laboratory cup-burner value (same as Halon 1301).

[c]10 percent safety factor above laboratory sphere value (same as Halon 1301).

SECTION IV
TOXICOLOGY OF CF_3I

A. INTRODUCTION

At the time CF_3I was announced as a potential near-term halon replacement in May 1993, little toxicological information was available on the chemical. What was known suggested that CF_3I was potentially low in toxicity (Reference 15). Since May 1993, a number of toxicological tests have been performed under a coordinated working group effort: acute inhalation modified limit, 15-minute rat LC_{50}, 4-hour acute inhalation studies, cardiac sensitization, gas uptake kinetics, quantitative structure activity relationship (QSAR) evaluation of the carcinogenic potential, and genetic toxicity screening tests. A discussion of each test and results for CF_3I are presented herein. Conclusions to date and recommendations for follow-on toxicity work are also presented.

B. ACUTE TOXICITY

Acute toxicity relates to the toxicity manifested within a relatively short time interval, usually on the order of minutes to days. Such toxicity is often the result of a single exposure. Inhalation studies performed with CF_3I indicate that it has a low acute toxicity. A modified acute inhalation "limit test," which assessed the effects of CF_3I in rats at some increment above the extinguishment concentration, was performed. In this case, four times the extinguishment concentration (approximately 12 percent) produced no adverse effects or lethality in rats exposed for 15 minutes (Reference 16). Animals experienced salivation and audible respiration during the exposure and recovered completely within 1 hour after the exposure.

The rat 15-minute LC_{50} (concentration to kill 50 percent of an animal test population) is equal to 27.4 percent by volume (Reference 17). Surviving rats experienced anesthesia but recovered quickly after exposure. Pathological examination after these acute studies revealed no abnormal findings. The rat 15-minute LC_{50} values for Halon 1211 and CFC-11 (a typical refrigerant) are, respectively, 20 percent (Reference 18) and 13 percent (Reference 19). Acute (4-hour) inhalation of CF_3I in rats at concentrations of 1.0 and 0.5 percent did not produce signs of toxicity during or following the exposures (Reference 20). Histological examinations of tissues from these exposed rats were normal.

Since the chemical contains an iodine atom, it is possible that exposure to CF_3I might interfere with thyroid function. To test this hypothesis, serological assays were performed following 4-hour exposure to CF_3I. These assays showed no biologically significant differences in thyroid hormones in circulating blood of CF_3I-exposed rats compared to control animals. The absence of lethality in rats exposed to 1 percent (10,000 ppm) CF_3I indicates that the compound could be classified as "practically non-toxic" (Reference 21).

Acute inhalation (6-hour) exposures (100, 400, and 800 ppm) were performed in rats to determine the uptake and metabolic degradation of CF_3I compared to Halon 1301 (Reference 22). These studies showed that CF_3I, as well as Halon 1301, was relatively insoluble in blood. This suggested that the chemical would not likely be transported from the lungs to blood and other tissues. In addition, based on pharmacokinetic modeling, minimal, if any, enzymatic metabolism of CF_3I or Halon 1301 occurred in rats. As a result of the favorable outcome from the acute studies cardiac sensitization testing was initiated.

C. CARDIAC SENSITIZATION

Cardiac sensitization is the term used for the phenomenon of the sudden onset of cardiac arrhythmias caused by a sensitization of the heart to epinephrine (adrenaline) in the presence of some concentration of a chemical. Cardiac sensitization (specifically leading to ventricular fibrillation) was first demonstrated in 1912 in cats exposed to chloroform in the presence of epinephrine, which without epinephrine was nonhazardous (Reference 23). Since then, cardiac sensitization has been demonstrated in man as well as laboratory animals.

When comparing concentrations necessary to elicit toxic responses such as anesthesia, cardiac sensitization, or lethality, cardiac sensitization occurs at a lower concentration than the other two endpoints. Therefore, regulatory and standard-making authorities have used cardiac sensitization thresholds as the criterion for determining acceptability for use in areas where human occupancy may occur. In addition, the phenomenon of cardiac sensitization is particularly important in firefighting because under the stress of the fire event, higher levels of epinephrine are secreted by the body, which increases the possibility of sensitization.

1. Cardiac Sensitization Testing Protocol

The experimental procedure used to investigate the cardiac sensitization potential of a chemical involves outfitting dogs with electrocardiographic (ECG) measurement devices and exposing the animals to a sequence of agent and epinephrine (Reference 24). Healthy male beagle dogs (generally 6 or more animals per exposure concentration), between the age of 1 to 2 years, are trained to stand in a cloth sling and to wear a snout mask. The dogs learn to accept venipuncture and ECG monitoring; thus, they are minimally stressed during the experiment.

The usual sequence of exposure is that the animal is monitored in a baseline condition without any intervention for 2 minutes (Table 7). Epinephrine is then intravenously infused to determine the effect of this catecholamine on the cardiac system. The dosage and time period for infusion vary slightly between laboratories; however, the levels of epinephrine given are always in the pharmacological rather than the physiological range. After approximately 5 minutes from the initial epinephrine administration, the agent is given as a continuous inhalation exposure either through a mask fitting over the dog's snout or in an exposure chamber. After a 5-minute agent exposure, epinephrine is administered ("epinephrine challenge") intravenously along with the continuous agent exposure. The animals is monitored for another 5 minutes to determine the effect of epinephrine and agent. This protocol is performed at increasingly higher doses until a "marked adverse response" occurs.

TABLE 7. PROTOCOL FOR TESTING CARDIAC SENSITIZATION IN DOGS.

Time, minutes	Procedure
0	Start ECG recording
2	Administer epinephrine dose
7	Start inhalation of test gas or air
12	Administer epinephrine challenge dose
17	Stop test gas inhalation; stop ECG recording

A "marked adverse response" is considered as the appearance of five or more multifocal ventricular ectopic beats or ventricular fibrillation (Reference 25). A "mild response" is described as an increase in the number of isolated abnormal beats (less than 5 consecutive beats) following the epinephrine challenge (second epinephrine administration). The threshold level is the lowest concentration at which cardiac sensitization occurs. No definitive rule exists indicating the number of animals that must experience a marked response to determine the threshold value. In most cases, even one animal experiencing a marked response constitutes establishment of a threshold value. This level is also called the Lowest Observable Adverse Effect Level (LOAEL). The highest concentration at which no marked responses occur is called the No Observable Adverse Effect Level (NOAEL). These values are used when determining safe exposure levels for humans. However, it is not known with certainty whether the LOAEL and NOAEL in dogs accurately correspond to these values in humans since a study directly comparing cardiac sensitization levels in dogs and humans is not available.

2. Cardiac Sensitization Test Results

Using the above protocol, the cardiac sensitization potential of CF_3I was determined in dogs. The NOAEL was measured as 0.2 percent (2000 ppm) and the LOAEL was 0.4 percent (4000 ppm).* At 0.2 percent, none of the six animals tested experienced adverted effects. At 0.4 percent, one dog was tested, and exposure to CF_3I resulted in ventricular fibrillation and death. These concentrations are similar to those that cause cardiac sensitization with CFC-11 (a widely used refrigerant), Halon 1211, and a number of other halon replacement agents (Table 8).

3. Interpretation of Cardiac Sensitization Results

Despite the fact that cardiotoxic thresholds are conservative for humans even in high-stress situations (Reference 26), regulatory and standard-making authorities are using results of cardiac sensitization tests to determine the acceptability of halon replacements for use in normally occupied total-flood applications. If the cardiac sensitization value (LOAEL for EPA or NOAEL for NFPA) is below the fire suppression design concentration, the candidate is not acceptable for use in normally occupied total-flood applications. Since the design concentration for CF_3I has been recommended to be 5.0 to 7.0 percent and the LOAEL is 0.4 percent, CF_3I is not suitable for occupied applications.

---

* Dodd, D., Personal communication, Mantech Environmental Technologies, Inc., WPAFB, OH, September 1994.

TABLE 8. COMPARISON OF CARDIAC SENSITIZATION VALUES.

Chemical	NOAEL, %	LOAEL, %	Reference
CF_3I	0.2	0.4	*
Halon 1211	0.5	1.0	27
HCFC-123	1.0	2.0	28
HCFC-124	1.0	2.5	26
HCFC-22	2.5	5.0	25
CFC-11	0.13	0.35	19

* Dodd, D., Personal communication, Mantech Environmental Technologies, Inc., WPAFB, OH, September†1994.

Because design concentrations are not typically thought of for streaming agents, the EPA uses models or air monitoring data to determine if the exposure level will exceed the cardiac sensitization threshold. During breathing zone personnel monitoring studies of other halon replacement agents, personnel were exposed to less than 0.1 percent agent concentration in simulated aircraft hangar exposures during discharge of 20- or 150-pound fire extinguishers in T-dock aircraft hangars (Reference 29) and in open pit, outdoor fire scenarios fought with 20- or 150-pound fire extinguishers (Reference 30). Another study showed firefighter breathing zone concentrations of less than 0.1 percent in real-fire flightline scenarios fought with 150-pound extinguishers using either Halon 1211, HCFC-123, or perfluorohexane (C_6F_{14}) (Reference 31). Accordingly, it is anticipated that in outdoor and T-hangar streaming scenarios similar to those indicated above, firefighter exposure would not exceed concentrations greater than the NOAEL for CF_3I of 0.2 percent.

Cardiac sensitization test results preclude the use of CF_3I in occupied areas where human occupancy is expected. In streaming applications, human occupancy is necessary to operate the manually discharged extinguishers. Accordingly, exposure to the agent is possible and a careful risk assessment must be performed, taking into account the toxic levels for the chemical and the typical exposure levels for the particular scenario. When calculating risks for chemical substances, a number of factors are usually considered. Variations in human sensitivity and animal-to-human extrapolations are two such factors. Often a factor of 10 is given to each of these uncertainties (i.e., 10 x 10 = 100). However, the cardiac sensitization test method "sensitizes" the test animals during the procedure as a result of the pharmacological doses of epinephrine administered. Therefore, most toxicologists concur that a 100-fold factor is not required. EPA currently does not consider safety factors because of the highly conservative nature of the cardiac sensitization test method. However, the Committee on Toxicology (COT) is currently determining whether safety factors are required for the cardiac sensitization test.

D. MUTAGENICITY TESTING STRATEGIES

Chemical carcinogenicity is usually the result of long-term consistent exposure to the chemical, generally doing manufacturing or servicing of equipment. A battery of genotoxicity screening tests are often performed to determine experimentally the mutagenicity of an agent. Positive mutagenicity results alert toxicologists to the possibility of carcinogenesis and indicate the need for subchronic exposure testing to develop industrial exposure standards. The "genotox" battery performed on CF_3I included the Ames test, the mouse lymphoma assay, and mouse micronucleus test. Although not performed on CF_3I , other genotox screens are available.

1. Ames Test

The Ames test, an *in vitro* ("in glass" or test tube) test for mutagenicity, and by implication, carcinogenicity, uses mutant strains of bacterium *Salmonella typhimurium* as a preliminary screen for carcinogenic potential (Reference 32). A number of assays comprise the Ames test, and positives indicate that a mutation in the genetic material has occurred. The mouse lymphoma test, also an *in vitro* screening test, uses cell cultures of mouse lymphoma cells. The mutagenic potential of a material is tested by observing the ability to confer resistance within this cell line to normally toxic agents. Mutations in the genetic material allow the cells to grow in the presence of other known toxic materials (purines, pyrimidines, or ouabain). Promutagens (mutagenic agents that require metabolic activation) can also be identified.

2. Mouse Micronucleus Test

The mouse micronucleus test, an *in vivo* ("in the living" or in live animals) test, determines the potential of a chemical to cause chromosome breakage or interference with normal cell division. The test entails exposing live mice to the test material, then removing premature red blood cells from the bone marrow, and observing the cells for the presence of chromosome fragments or the lack of signs of normal cell division. This test is not considered the most sensitive test for chromosomal aberrations (Reference 33).

3. Other Screening Tests

Other *in vitro* tests that yield information on the carcinogenic potential of an agent, but which have not been performed on CF_3I, include the following:

(1) Unscheduled DNA Synthesis (UDS) test, which involves the exposure of cultured hepatocytes (liver cells) to the test chemical and monitoring the repair of DNA following DNA damage by a mutagen.

(2) Sex-linked recessive mutation test for mutagenicity, which utilizes *Drosophila melanogaster* (fruit fly) males with a marker (yellow body) on the X chromosome.

(3) Sister chromatid exchange test, which can also be an *in vivo* test, detects DNA alkylating agents in Chinese hamster ovary cells.

(4) The dominant lethal (rodent) test, which is an *in vivo* test that assesses the ability of a suspected mutagen, which has shown positive in an *in vitro* screen, to cause dominant lethal mutations in rats, mice, or hamsters. Male rodents are treated with the test substance and are then mated to groups of females over several weeks to test for effects occurring at all stages of sperm formation. Following sacrifice, the females are evaluated for a number of fertility indices.

4. 90-Day Subchronic Toxicity Test

While the 90-day subchronic toxicity test is not specific for carcinogenicity or mutagenicity endpoints, it is a definitive study that gives possible indications of carcinogenic potential. This *in vivo* test in whole animals allows histological examination of tissues to determine whether neoplastic (uncontrolled cell proliferation) changes occurred due to chemical exposure. The subchronic test is a lower cost means of identifying carcinogenic potential than a lifespan chronic study. Nonetheless, the final determinant of carcinogenicity is a whole animal chronic bioassay. Chronic toxicity tests are conducted over the greater part of the animals lifespan starting at weaning (1.5-2 years in mice and 2 or more years in rats). The principal endpoint is tumor formation, as determined by histological exam.

5. Carcinogenicity Test Results to Date for CF_3I

a. Carcinogenicity Modeling Results

The carcinogenic potential of CF_3I was first evaluated using robust quantitative structure activity relationships (QSAR) (Reference 33). The well-known TOPKAT program was used to predict the carcinogenicity of CF_3I based on a set of chemical structure descriptors. Results were compared with Halon 1301. Although the comparison database used to model the carcinogenic potential was small, the estimates of carcinogenicity for CF_3I and Halon 1301 were given as 0.0, meaning they were predicted to be non-carcinogenic.

b. Genotox Results

A genotox battery was performed on CF_3I and yielded mixed results.* Four of 5 Ames assays were positive; the mouse micronucleus test was positive; and the mouse lymphoma test was unequivocally negative.† Since these screening tests are best for finding agents that cause direct DNA damage and since not all the tests indicated positive results, it is highly unlikely that CF_3I causes direct DNA damage as its primary effect.

6. Interpretation of Carcinogenicity Results

For years the predictive nature of short-term *in vitro* tests for carcinogenicity has been questioned (Reference 34). The degree to which the *in vitro* results correlate with carcinogenic or mutagenic activity in whole animals resulting in actual tumor formation largely depends on chemical class. For fluorinated derivatives of the hydrocarbon series, the correlation has not proved to be exact. The fact that the results of the genotox screen were not consistent is not typical, but not highly unusual either. A number of other halocarbons being considered as CFC and halon replacements have had similar genotox screens performed on them. In addition, a number of these chemicals have also had chronic bioassays performed. Table 9 presents these results of the genotox screens as well as the chronic tests.

* Dodd, D., Personal communication, Mantech Environmental Technologies, Inc., WPAFB, OH, September 1994.

† Dodd, D., Personal communication, Mantech Environmental Technologies, Inc., WPAFB, OH, September 1994

As seen in Table 9, all but one chemical have positive and negative results on one or more genetic toxicity screening tests. In follow-up chronic studies, the majority of the chemicals proved not to be carcinogenic, despite the mixed results in the screening test battery. For HFC-23, mixed results were obtained in the genetic toxicity screening tests, but a chronic study was never performed. Nonetheless, the EPA has approved its use as a halon replacement (Reference 35). For HFC-134a, all of the screening tests were negative, whereas in the chronic study, male rats developed benign testicular tumors. (Note: The tumors are specific to this species.) HCFC-123, on the other hand, had mixed results in the *in vitro* screen tests and showed benign tumor formation in the liver, pancreas, and testes in the chronic study. Despite the incidence of benign tumor formation, the EPA has approved the use of HFC-134a and HCFC-123 as refrigerants and halon replacements (Reference 34). In the case of HFC-134a use in refrigerant applications, the potential for general population exposure is possible since this chemical is being used in automobile air conditioners where the equipment is serviced and maintained on a routine basis.

Table 9 demonstrates that genotox screens do not necessarily accurately predict the carcinogenic potential of fluorocarbon chemicals. Also, safe exposure guidelines and handling procedures can be established to allow the use of chemicals, despite evidence of tumor formation in animal species. These guidelines and procedures have been derived for HFC-134a and HCFC-123. Moreover, regulatory approval can be granted for chemicals that have not had full carcinogenicity testing (HFC-23).

E. SUMMARY

The amount of toxicological information on CF_3I has increased dramatically in a relatively short time span due to a coordinated working group effort. Within a year, short-term (acute) inhalation studies, cardiac sensitization tests, and preliminary genetic toxicity tests have been performed (Table 10). Acute inhalation studies revealed that CF_3I has a low order of toxicity, with an LC_{50} value for a 15-minute exposure in rats equal to 27.4 percent. This is better than Halon 1211 or CFC-11 (a common refrigerant), which have 15-minute LC_{50} values equal to 20 and 13 percent, respectively. For these and other toxicological values, higher numbers mean it takes more chemical to produce the toxic result. Consequences of short-term exposures to CF_3I included anesthesia, salivation, and audible breathing. Blood chemistry measurements showed no abnormal results.

TABLE 9. GENOTOX SCREEN AND CHRONIC TEST RESULTS ON CFC AND HALON REPLACEMENTS.

Chemical	Genotox Results[a]	Chronic Results[a]
CF_3I (Fire extinguishant)	Positive (4 out of 5 Ames Test and mouse micronucleus assay)[b] Negative (Mouse lymphoma)[b]	Not known
HFC-23 (Fire extinguishant and refrigerant)	Positive (*Drosophila melanogaster* sex-linked recessive mutation test/References 36, 37) Negative (Ames Test/Reference 35)	Not known
HFC-152a	Positive (*Drosophila melanogaster* sex-linked recessive mutation test/References 37, 38) Negative (Ames Test/Reference 35)	Negative in rats (Reference 38)
HFC-134a (Refrigerant)	Negative (Ames Test, BHK21 Cell Transformation Test/Reference 35; Chinese hamster ovary chromosome aberration test, *in vivo* rat lymphocytes, mouse micronucleus/Reference 26)	Benign tumors in rats (testes) (Reference 26)
HCFC-22 (Refrigerant)	Positive (Ames Test/Reference 35) Negative (BHK21 Cell Transformation Test/Reference 35)	Negative in rats (Reference 35)
HCFC-123 (Fire extinguishant and refrigerant)	Positive (Human lymphocyte chromosome aberration/ Reference 26) Negative (Ames Test, Chinese hamster ovary chromosome aberration test, *in vivo* rat lymphocytes, mouse micronucleus/Reference 26)	Benign tumors in rats (liver, pancreas, testes) (Reference 26)
HCFC-143a	Positive (Ames Test/Reference 35) Negative (BHK21 Cell Transformation Test/Reference 35)	Negative in rats (Reference 35)
HCFC-142b	Positive (Ames Test, BHK21 Cell Transformation Test/Reference 35) Negative (Rat micronucleus test, rat dominant lethal test/Reference 39)	Negative in rats (Reference 35)

[a]No data available for Halons 1211 and 1301; mutagenicity for these compounds is unknown.

[b]Dodd, D., Personal communication, Mantech Environmental Technologies, Inc., WPAFB, OH, September 1994.

TABLE 10. SUMMARY OF TOXICOLOGICAL INFORMATION ON CF_3I.

Toxicological Property	Value, %, or Status
LC50, rat, 15-minute	27.4
Cardiac Sensitization	
NOAEL	0.2
LOAEL	0.4
Carcinogenicity/Mutagenicity	
Genotoxicity Tests	
Ames Test	4 out of 5 positive
Mouse Lymphoma	Unequivocally negative
Mouse Micronucleus	Positive (after reanalysis)
Subchronic Test, 13-wks(90-days)	Unknown (in progress at Armstrong Labs, started October 19, 1994)
Developmental Toxicity	Unknown (testing planned at Armstrong Labs)
Degradation Byproducts	HF, HI, CO (concentrations similar to Halon 1301)

Cardiac sensitization, a potential adverse effect from exposure to halons and CFCs, is the production of irregular heart beats and sometimes cardiac arrest in response to exposure of air-borne chemicals and intravenous adrenaline. Cardiac sensitization measurements indicated that the NOAEL, the highest concentration at which no detrimental responses occur, is 0.2 percent and the LOAEL, the lowest concentration at which negative effects do occur, is 0.4 percent. These cardiac sensitization values are similar to CFC-11 (NOAEL = 0.13 and LOAEL = 0.35), Halon 1211 (NOAEL = 0.5 and LOAEL = 1.0), and other halon replacement agents (HCFC-123, NOAEL = 1.0 and LOAEL = 2.0; HCFC-124, NOAEL = 1.0 and LOAEL = 2.5). Since the expected design concentration for CF_3I will be in the range of 5 to 7 percent, this cardiac toxicity profile will preclude its use for total-flood applications in normally occupied areas. However, CF_3I remains a potentially promising alternative for total flooding in unoccupied areas and for streaming applications.

The potential for carcinogenic effects of CF_3I has not been completely evaluated to date. The screening results in test tube assays are mixed; some tests indicate the potential for genetic alterations, while others suggest no changes leading to cancer-causing results. Mixed results on these types of screening tests are not unusual. Most of the halon and CFC replacements also exhibit positive and negative results. For example, HFC-23, HFC-152a, HFC-143a, HCFC-22, HCFC-123, and HCFC-142b had mixed results on screening tests. Only HFC-134a was completely negative in test-tube screening assays. In addition, these screening assays do not always accurately predict the tumor-forming potential when tested in live animals. Therefore, the available information on CF_3I is insufficient to conclude that its use in firefighting applications is unsafe. Further testing on CF_3I will need to be performed to evaluate the carcinogenic potential in live animals.

F. CONCLUSIONS AND RECOMMENDATIONS

1. Conclusions

The toxicity resulting from a chemical is generally a consequence of short-term and/or long-term exposures. In firefighting applications, short-term high concentration exposures occur during the use scenarios, while long-term, low concentrations result during manufacturing and servicing of equipment. Firefighting exposures are difficult to control, but can be limited through protective equipment and proper use training. Manufacturing and servicing exposures are controllable through training and protective equipment.

The amount of toxicological information on CF_3I has increased dramatically in a relatively short time span. Within a year, acute inhalation studies, cardiac sensitization tests, and preliminary genetic toxicity tests have been performed. CF_3I is a highly promising replacement in both total flooding (of unoccupied areas) and streaming applications. The potential for carcinogenic effects has not been completely evaluated to date.

2. Recommendations

a. Further testing (90-day subchronic) should be completed to evaluate preliminarily the carcinogenic potential in whole animal systems before CF_3I is eliminated as a halon replacement.

b. The relationship between cardiac sensitization in dogs humans should be investigated to determine the appropriateness for using the cardiac sensitization threshold for setting exposure limits.

SECTION V
GLOBAL ENVIRONMENTAL STATUS

A. ATMOSPHERIC LIFETIME

CF_3I and other FICs have a strong tendency to photolytically decay in visible light, resulting in a very short atmospheric lifetime. The atmospheric lifetime of CF_3I in the atmosphere depends on the rate of its photolysis. An accurate temperature dependence of the absorption cross section was essential to determine the accurate global environmental impact of CF_3I. Several organizations readily generated cross sections for CF_3I (References 5 and 40). The following decomposition mechanisms have been proposed:

$$CF_3I + h\nu \rightarrow CF_3 + I \text{ (days)}$$
$$CF_3I + OH \rightarrow CF_3 + HOI$$
$$CF_3 + O_2 \leftrightarrow CF_2O + FO \quad (< \text{seconds})$$
$$CF_2O + H_2O \rightarrow CO_2 + 2\,HF \text{ (hours)}$$

Based upon model results to date, the atmospheric lifetime of CF_3I is less than 1 day (Reference 41). The atmospheric lifetime for releases at higher altitudes is thought to be significantly shorter.

B. OZONE-DEPLETION POTENTIAL (ODP)

The most current estimated ODP for CF_3I is less than 0.008 and more likely 0.0001 (Reference 41) relative to CFC-11.

If CF_3I were to be released directly into the stratosphere during aircraft releases, the ODP would be considerably higher, due to the direct injection of the iodine atom. Therefore, data have been collected on atmospheric releases of Halon 1301 to gain insight into possible high altitude releases of CF_3I as a future aircraft fire suppression agent. Based on figures compiled by the US Air Force, US Navy and commercial aircraft industry the current release of Halon 1301 during flight is less than 1000 pounds annually, of which 160 pounds are released at altitudes above 30,000 ft. Because this quantity is so low, expected high altitude releases of CF_3I, if used on aircraft, would not be considered a significant threat to the ozone layer (Reference 40).

C. GLOBAL WARMING POTENTIAL (GWP)

Due to the short atmospheric lifetime of the chemical and the photolytic decomposition mechanism proposed above, the GWP is less than 1×10^{-3} relative to CFC-11. Therefore, the 20-year time horizon GWP of CF_3I is extremely small (less than 5) relative to CO_2.

D. CONCLUSIONS

The global environmental characteristics of CF_3I were recently summarized in a paper from NOAA (Reference 41), which states that

> . . . the extremely short lifetime of CF_3I greatly limits its transport to the stratosphere when released at the surface, especially at midlatitudes, and the total anthropogenic surface release of CF_3I is likely to be far less than that of natural iodocarbons such as CF_3I on a global basis. It is highly probable that the steady-state ozone depletion potential (ODP) of CF_3I for surface releases is less that 0.008 and more likely below 0.0001. Measured infrared absorption data are also combined with the lifetime to show that the 20-year global warming potential (GWP) of this gas is likely to be very small, less than 5. Therefore this study suggests that neither the ODP nor the GWP of this gas represents significant obstacles to its use as a replacement for halons.

SECTION VI
COMMERCIALIZATION PROCESS

A. MANUFACTURABILITY

There are two declared large-scale US manufacturers of CF_3I and other FICs: Pacific Scientific and West Florida Ordnance (WFO). Pacific Scientific has recently increased production from 50 to 500 pounds/day and has built a new plant in Oklahoma that will have an ultimate 3-5 million pounds/yr capacity. WFO has a capacity of 100 pounds of FICs/8-hour shift and, depending on demand, could operate multiple production shifts. WFO is in the process of constructing a new plant that could manufacture a variety of FICs. Pacific Scientific is marketing CF_3I under the tradename Triodide®, while WFO is using the tradename Iodoguard®. There are also several US producers of laboratory quantities of FIC chemicals, as well as production locations in Russia and Japan.

B. US EPA SIGNIFICANT NEW ALTERNATIVES POLICY LISTING

The US Clean Air Act Amendments of 1990, Title VI, Section 612 requires the US EPA to enact regulations making it unlawful to replace CFCs or halons with any replacement that may impact human health or the environment and requires EPA to publish lists of acceptable and prohibited substances. This listing is known as the Significant New Alternatives Policy (SNAP) Program List.

A Significant New Alternatives Policy (SNAP) application was submitted to the US Environmental Protection Agency (EPA) on behalf of Pacific Scientific for CF_3I under the tradename Triodide® in May 1994 for total-flood application in unoccupied spaces. Triodide® was proposed acceptable for unoccupied areas in August 1994 (Reference 42). In September 1994, a SNAP application was submitted on behalf of Pacific Scientific for CF_3I under the tradename Triodide® for use in streaming applications. The streaming application submittal is awaiting EPA action.

C. OTHER LISTINGS OR APPROVALS

At this time, the NFPA has not listed CF_3I for firefighting or explosion protection uses. No fire suppression systems, equipment, or component listing or approvals are available from Underwriter's Laboratories, Inc. (UL) or Factory Mutual, Inc. (FM) at this time with CF_3I. Pemall, Inc., in New Jersey has submitted a hand-held CF_3I fire extinguisher for UL testing. Currently, Fire Control Systems in Canada has submitted a pre-engineered system for UL Canada approval. Submission of additional equipment/systems is expected in the near future.

SECTION VII
STABILITY TESTING

A. INTRODUCTION

Under the requirements of the CF_3I Feasibility Study workplan developed by the CF_3I Working Group, it was necessary to obtain preliminary stability data for CF_3I. The stability testing was conducted at NIST and NMERI using entirely different sets of conditions. The NMERI test protocol was based on ASTM Standards and also incorporated materials compatibility tests. The stability test procedure consisted of a weekly Fourier transform infrared (FTIR) spectrometer analysis of the volatilized liquid phase of the chemical, with specific focus on determining the HF, CO_2, CO, and C_2F_6 content. Within the detection limits of the equipment utilized in the experiments, no breakdown of the agent was detected during the 180-day test period. During the testing, one accidental short-duration excursion to 116 °C (240 °F) produced HF peaks of about 100 ppm in the sample containing water vapor. The NIST testing, conducted as part of the USAF aircraft engine nacelle and dry bay halon replacement program, detected evidence of decomposition at around 149 °C (300 °F). Analysis of the data indicates that CF_3I appears to undergo degradation between 77 and 149 °C (170 and 300 °F).

B. TEST MATRIX AND TEST PROCEDURE

The stability test series consisted of 10 cylinders of CF_3I in combination with air, N_2, and water (Table 11) stored under two different temperatures. The cylinders used in the tests were made of stainless steel with high quality valves and pressure gauges.

TABLE 11. CF_3I STABILITY TEST MATRIX.

Sample	27 °C (80 °F)	77 °C (170 °F)
CF_3I neat	X	X
+ N_2 300 psi	X	X
+ N_2 300 psi, + 0.5% H_2O	X	X
+ N_2 600 psi	X	X
+ Air 300 psi	X	X

A Perkin Elmer System 2000 FTIR was used to record spectral data during the tests using potassium bromide (KBr) windows with a path length of 10 cm. A nitrogen purge was conducted before each test to obtain a clean background. The 10 sample cylinders were tested weekly at room temperature, and a spectrum for each sample was taken as outlined below.

- The sample cylinder was removed from the oven and allowed to cool to room temperature.
- The weight and pressure of the cylinders were measured and recorded.
- One sample chamber was attached to the cylinder.
- The cylinder was then attached upside down to a sample cell.
- The sample chamber was evacuated under house vacuum after the sample cylinder was attached.
- A background spectrum was taken while a N_2 purge was running.
- A gas sample was "burped" into the sample chamber and released into the sample cell where it was scanned by the FTIR.
- The weight of the cylinder and its pressure were recorded after being scanned by the FTIR.
- The cylinder was placed back into the oven.
- The FTIR scan was analyzed for HF and other abnormal absorbances (Figure 3).

During the FTIR scan mode, the instrument was set to perform five scans of each expanding gas sample. The five scans were averaged into one scan and saved as a sample spectrum. A new background was run prior to the next test in order to ensure a complete purge of the sample cell after the sample spectrum was saved.

C. RESULTS AND OBSERVATIONS

A CF_3I reference spectrum (Figure 3) was used for data analyses and the different spectra were obtained by subtracting the reference spectrum from each of the sample spectra. Throughout the entire six-month test period, there were no observable spectral changes under any of the sampling conditions shown in Table 11.

A controller failure caused an accidental excursion to 116 °C (240 °F) for the five 77 °C (170 °F) samples. One of the CF_3I air sample cylinders showed significant decomposition. A strong HF peak was exhibited in the sample spectrum. The concentration of HF in this cylinder

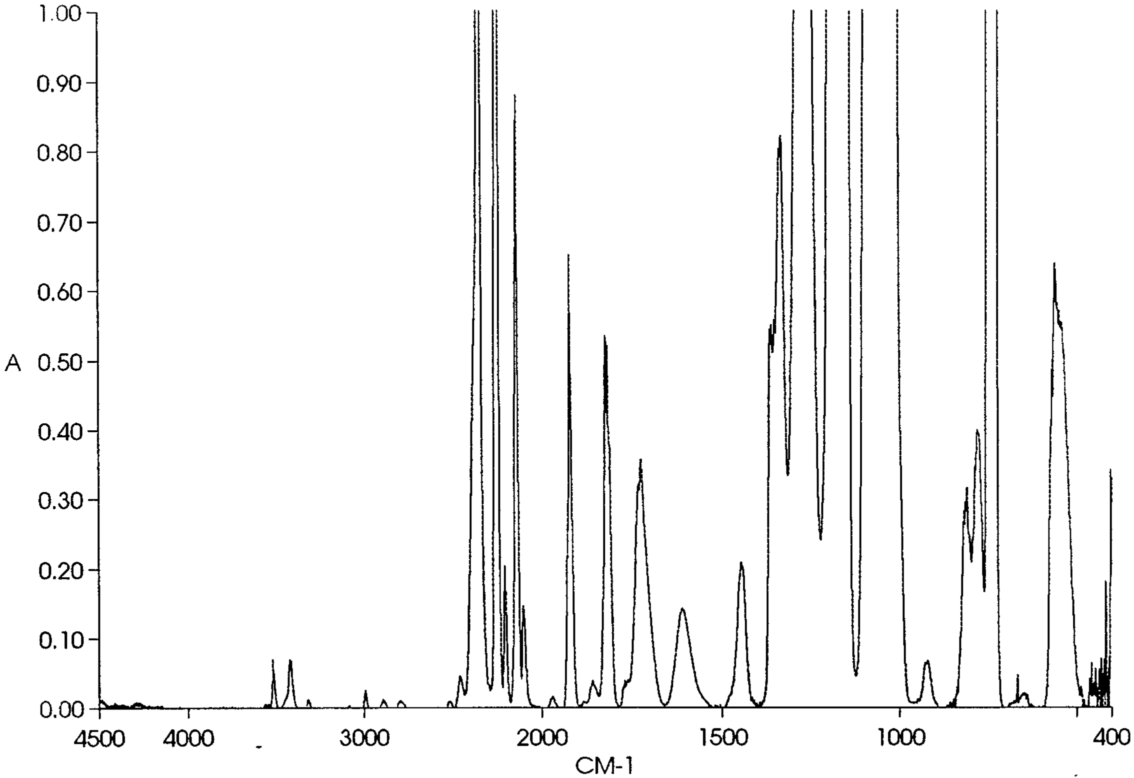


Figure 3. FTIR Reference Scan for CF_3I.

decreased over time, and it became undetectable by the end of the test series. It was assumed that the HF had been absorbed into the wall of the cylinder. Note, the bond vibrations of hydrogen iodide (HI) are not detectable with the FTIR due to the low dipole moments.

D. STABILITY TESTING AT NIST

The NIST testing, conducted as part of the USAF Wright Laboratory's aircraft engine nacelle and dry bay halon replacement program, detected evidence of decomposition at around 149 °C (300 °F). Analysis of the data indicates that CF_3I appears to undergo degradation between 77 and 149 °C (170 and 300 °F).

E. CONCLUSIONS

As with some other issues pertaining to CF_3I, comparable stability data for Halon 1301 is unavailable; it is therefore difficult if not impossible to determine the stability of FICs relative to Halon 1301. For nominal long-term storage conditions, it would appear that CF_3I is stable inside sealed cylinders. For severe operating conditions such as would be encountered in the vicinity of a fighter aircraft auxiliary power unit (APU), where temperatures could exceed 177 °C (350 °F) for short periods of time, CF_3I stability may not prove to be adequate. It should be noted that the behavior of Halon 1301 under this type of severe scenario is largely unknown.

From the six-month (180-day) CF_3I stability test results it is concluded that little or no degradation occurred at 27 or 77 °C (80 or 170 °F) under a variety of conditions. Under specific conditions, a significant degradation of CF_3I can occur at temperatures approximating 116 °C (240 °F). HF was the major decomposition product detected.

SECTION VIII
MATERIALS COMPATIBILITY TESTING

A. INTRODUCTION

Under the requirements of the CF_3I Feasibility Study workplan developed by the CF_3I Working Group, it was also necessary to obtain preliminary materials compatibility data for CF_3I and major materials of construction used in the manufacture of fire extinguishing systems. The primary purpose of this study was to determine whether specific materials compatibility factors exist that would make the use of CF_3I unfeasible.

Due to the limited amount of material available, the temperature range of interest, and the need for more quantitative data, the ASTM "Standard Test Method for Metal Corrosion by Halogenated Organic Solvents and Their Admixtures" (ASTM D 2251-85) (Reference 43) was modified. These modifications were made using the general ATSM Standard Practices: ASTM G1-90 "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens," and ASTM G 31 "Standard Practice for Laboratory Immersion Corrosion Testing of Metals."

The test materials were suggested by the CF_3I Working Group members based on their common use in the components of fire suppression systems. These materials were: nitrile-butadiene rubber (NBR or Buna N); neoprene; ethylene-propylene diene monomer (EPDM), which are used in seals and gaskets; and metals (stainless steel, carbon steel, aluminum, brass, and copper) used in storage cylinders, delivery tubes, etc. The required test temperatures were 27 and 77 °C (80 and 170 °F). Samples were evaluated at intervals of 30, 90, and 180 days.

B. METAL SAMPLE ANALYSIS

1. Test Matrix and Test Procedure

Four types of metal materials were tested for compatibility with CF_3I: 21-6-9 stainless steel (nitronic 40); carbon steel; 7075 aluminum; and casting brass. Eighteen samples of each metal type were divided into two groups of nine samples each and, after being cleaned and weighed, were placed in 27 and 77 °C (80 and 170 °F) ovens. The procedure used for the cleaning and preparation of the corrosion test metal specimens is fully described in the ASTM Standard Practice ASTM G1-90, "Standard Practice for Preparing, Cleaning, and Evaluating

Corrosion Test Specimens" (Reference 43). In accordance with the procedure's specifications, the following method was used to prepare metal specimens:

(1) Ultrasonic cleaning using a 2 percent solution of Alconox detergent in hot tap water for 2 minutes.
(2) Rinse with tap water.
(3) Abrasive cleaning with 600 grit sandpaper for 30 seconds each side.
(4) Ultrasonic cleaning in distilled water for 1 minute.
(5) Rinse with tap water.
(6) 30-second ultrasonic cleaning in distilled water.
(7) Rinse with methanol.
(8) Dry in oven at 75 °C (167 °F) for 30 minutes.
(9) Cool in dessicator to room temperature.

All test specimens were sealed in individual autoclaves after being cleaned (Figure 4). CF_3I was distilled onto the specimens and sample cylinders were stored at 27 and 77 °C (80 and 170 °F) for the required period. At the end of each required testing period, the sample cylinders were taken out of the oven and the CF_3I was distilled off. The corrosion rate of the metal samples were calculated after they had been weighed and cleaned according to the ASTM procedures:

<u>Aluminum</u>— According to ASTM G1-90 C.1.1:

(1) 54 mL H_2SO_4 diluted to 1 liter
(2) Digested 5 minutes boiling at 94-95 °C
(3) Rinsed thoroughly with distilled water
(4) Rinsed thoroughly with methanol
(5) Placed in oven at 77 °C (170 °F) for 15 minutes
(6) Allowed to cool in dessicator before weighing

<u>Carbon Steel</u>— According to ASTM G1-90 C.3.5:

(1) 500 ml HCl + 3.5 g Hexamethylene tetramine diluted to 1 liter
(2) Digested 10 minutes at 25 °C (77 °F)
(3) Rinsed, dried, cooled as above

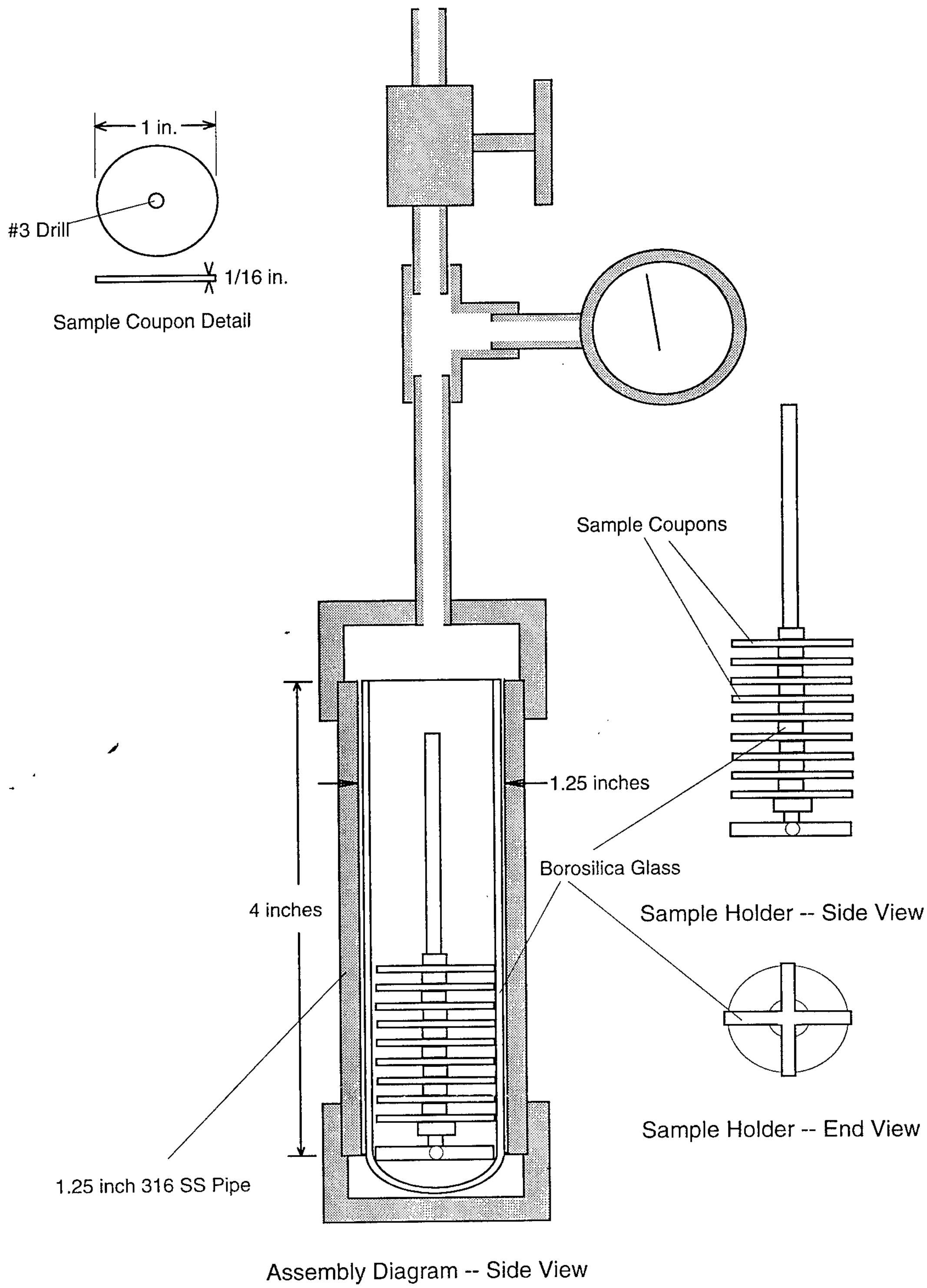


Figure 4. Materials Compatibility Test Apparatus.

Stainless Steel— According to ASTM G1-90 C.7.1:

(1) 100 ml HNO_3 diluted to 1-liter
(2) Digested 20 minutes at 60 °C (140 °F)
(3) Rinsed, dried, cooled as above

Brass— According to ASTM G1-90 C.2.1

(1) 50 ml H_3PO_4 + 20 g CrO_3 diluted to 1 liter
(2) Digested 30 minutes at 50 °C (122 °F)
(3) Rinsed thoroughly with distilled water
(4) Brushed 10 seconds each side with soft-bristled toothbrush
(5) Digested 4 minutes more at 50 °C (122 °F)
(6) Rinsed, dried, cooled as above

2. Results and Observations

Preliminary results and observations are shown in Table 12. Table 12 shows the numerical results and observable changes obtained from the 30, 90, 180-day material compatibility tests. Although slight modifications on surface appearance were noted, measured analyses of actual corrosion rates indicate at most 0.03 mm/yr; however, in the carbon steel case, the contaminants may have greatly increased corrosion rates.

C. RUBBER SAMPLE ANALYSIS

1. Test Matrix and Test Procedure

Three types of rubber were tested for compatibility with CF_3I: Buna N; neoprene; and EPDM. Eighteen samples of each rubber type were used during the 180-day test and each was cleaned following the procedure shown below:

(1) Cleaned in ultrasonic cleaner with Alconox Solution (10 ml/2000 ml H_2O) for 2 min.
(2) Rinsed the coupon with tap water.
(3) Thirty seconds in ultrasonic cleaner with deionized water.
(4) Rinsed the sample with CH_3OH.
(5) Stored in dessicator until commencement of test.

TABLE 12. RESULTS AND OBSERVATIONS OF THE METALS MATERIALS COMPATIBILITY TESTING.

Metal	Average Wt. Change, g		Corrosion Rate, mm/yr		Observations
	27 °C (80 °F)	77 °C (170 °F)	27 °C (80 °F)	77 °C (170 °F)	
Stainless Steel					
30-Day	0.0002	0.0006	-0.0004	-0.0006	No observable changes
90-Day	0.0000	0.0006	0.0002	-0.0002	No observable changes
180-Day	0.0005	0.0003	0.0004	0.0001	No observable changes
Carbon Steel					
30-Day	0.0004	0.0002	0.0025	0.0023	Both temperature samples had small spots of dark opaque substance.
90-Day	0.0188	0.0002	0.0098	0.0010	80 °F Sample coated with a black substance; significant weight gain. 170 °F Sample had small spots of a dark substance.
180-Day	0.0158	0.0327	0.0134	0.0288	80 °F Sample coated thickly with a rust substance; significant weight gain. 170 °F Sample coated thickly with a black substance; significant weight gain.
Brass					
30-Day	0.0004	0.0041	-0.0001	0.0040	80 °F Sample no observable change. 170 °F Sample covered with reddish-brown substance; significant weight gain.
90-Day	0.0005	0.0051	0.0001	0.0022	80 °F Sample no observable change. 170 °F Sample covered with reddish-brown substance; significant weight gain.
180-Day	0.0008	0.0033	0.0001	0.0031	80 °F Sample no observable change. 170 °F Sample covered with reddish-brown substance; significant weight gain.
Aluminum					
30-Day	0.0013	0.0019	0.0027	0.0012	80 °F Sample no observable change. 170 °F Sample had small white spots.
90-Day	0.0007	0.0004	-0.0001	0.0002	80 °F Sample no observable change. 170 °F Sample had small white spots.
180-Day	0.0026	0.0003	0.0022	0.0002	80 °F Sample no observable change. 170 °F Sample had small white spots.

After the cleaning procedure, the samples were weighed on an analytical balance. The inner diameter, outer diameter, and thickness of each sample were measured with a micrometer. A durometer Type A, calibrated in accordance with ASTM D2240, was used for the sample hardness measurement.

The 18 samples of each rubber type were divided into two groups of 9 samples each and designated "A" or "B". After placing the rubber coupons in individual autoclaves (Figure 4) filled with CF_3I, the "A" series were placed in the 27 °C (80 °F) oven and the "B" series in the 77 °C (170 °F) oven. Three samples of each series were evaluated at intervals of 30, 90, and 180 days.

2. Preliminary Results and Observations

The results and observations of the seal and gasket materials compatability tests are shown in Table 13. A summary of the results are presented in Table 14.

One month after beginning the test, samples were removed from the 27 and 77 °C (80 and 170 °F) ovens. The CF_3I was distilled into a temporary storage cylinder. When the cylinders were opened, the inner cells (glass part) were found to be coated with a brown viscous substance. This was suspected to be the light oil that is typically used as a plasticizer in standard commercial rubber formulations. The oil was removed by swirling the samples on a glass stick in acetone solution. On subsequent sample removals (after 3 months and 6 months), there was a marked decrease in the amount of oily residue as the plasticizer was depleted from the rubber. Even though it was still necessary to rinse the samples with acetone, the weight gain was smaller as was the difference in dimensions seen in Table 13.

The inner diameter, outer diameter, thickness, and hardness of the samples were measured after each of them had been blow-dried. In the course of weighing the rubber samples, it was noticed that they continued to lose weight as trapped CF_3I outgassed from the samples. Therefore, two measurements of each sample were taken. The samples were weighed within 30 minutes after the agent was removed and then again after being stored in a dessicator at room temperature for 24 hours. In comparing the results, the minimum absorption occurred with the EPDM rubber samples. All polymers showed little or no degradation and increased hardness during the 6-month testing period.

TABLE 13. RESULTS AND OBSERVATIONS OF THE ELASTOMER MATERIALS COMPATIBILITY TESTS.

Material Type	Immediate Measurement % Vol. Change	Immediate Measurement % Wt. Change	Immediate Measurement % Hardness Change	24-Hour Measurement % Vol. Change	24-Hour Measurement % Wt Change	24-Hour Measurement % Hardness Change
Buna N						
"A" Series (27 °C/80 °F)						
30-Day	10.6	40.2	-9.9	-18.6	-12.4	40.4
90-Day	12.5	49.0	-9.4	-22.4	-15.9	48.5
180-Day	14.9	71.3	-11.7	-25.2	-18.8	56.7
B" Series (77 °C/170 °F)						
30-Day	27.3	72.5	-12.3	-15.4	-9.5	38.0
90-Day	-1.1	28.4	17.0	-21.5	-11.6	55.6
180-Day	4.6	46.2	28.7	-21.8	-8.7	63.7
EPDM						
"A" Series (27 °C/80 °F)						
30-Day	-16.7	6.6	16.2	-16.0	-24.5	38.6
90-Day	-28.4	-25.8	32.4	-28.6	-29.1	37.6
180-Day	-28.8	-16.8	33.8	-30.5	-29.5	39.5
"B" Series (77 °C/170 °F)						
30-Day	-15.0	8.6	16.7	-26.3	-23.1	37.6
90-Day	-26.3	-16.4	33.8	-28.7	-28.0	37.1
180-Day	-24.8	-0.4	31.4	-31.1	-28.5	39.5
Neoprene						
"A" Series (27 °C/80 °F)						
30-Day	26.1	63.0	-19.0	-16.4	-12.1	19.6
90-Day	-3.7	16.7	-4.8	-23.8	-17.0	27.5
180-Day	12.0	58.9	-11.6	-27.3	-16.6	32.3
"B" Series (77 °C/170 °F)						
30-Day	28.5	66.6	-20.1	-15.6	-8.3	24.9
90-Day	-10.6	15.4	27.0	-21.5	-4.0	43.4
180-Day	-4.9	32.2	23.3	-19.6	-1.7	44.4

TABLE 14. SUMMARY OF CF_3I ELASTOMER MATERIALS COMPATIBILITY TESTS.

Material	% Volume Change	% Weight Change	% Hardness Change
	Immediate Measurement		
Buna N	-1.1 to 27.3	28.4 to 72.5	-12.3 to 17.0
EPDM	-28.8 to -15.0	-25.8 to 8.6	16.2 to 33.8
Neoprene	-10.6 to 28.5	15.4 to 66.6	-20.1 to 27.0
	24-Hour Measurement		
Buna N	-25.2 to -15.4	-18.8 to -9.5	38.0 to 63.7
EPDM	-31.1 to -16.0	-29.5 to -23.1	39.5 to 37.1
Neoprene	-27.3 to -15.6	-17.0 to -1.7	19.6 to 44.4

D. CONCLUSIONS

No compatibility problems are anticipated between CF_3I and the materials of construction used in fire extinguishing systems. The material compatibility test results show little or no metal corrosion with pure CF_3I at both 27 and 77 °C (80 and 170 °F). Contaminants may greatly increase the metal corrosion rates especially for carbon steel. All three polymers showed very little or no degradation or change in hardness under both test temperatures. The minimum absorption occurred with the EPDM rubber samples.

SECTION IX
CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

Several physical and chemical properties of CF_3I have been determined. It is a dense liquid (liquid density = 2.10 g/ml) with a boiling point (-22.4 °C) somewhat higher than Halon 1301. Based upon confirmation of previous laboratory data (cup-burner value = 3.1 percent, propane flammability peak = 6.5 percent) and results of small and intermediate-scale testing to date, CF_3I is considered to be an isovolumetric halon replacement for Halon 1301 in most applications. The recommended fire suppression and propane inertion design concentrations are 5.0 and 7.0 percent, respectively, same as Halon 1301. CF_3I has also been shown to be a isovolumetric replacement for Halon 1211 in streaming applications.

The toxicological information on CF_3I has increased dramatically in a relative short time. Within a year, acute inhalation studies, cardiac sensitization tests, and preliminary genetic toxicity tests have been performed. Acute inhalation studies revealed that CF_3I has a low order of toxicity with a 15-minute LC_{50} equal to 27.4 percent. Effects during acute studies included anesthesia, salivation, and audible respiration. Serum chemistry measurements showed no abnormal results. Cardiac sensitization measurements indicated that the NOAEL is 0.2 percent and the LOAEL is 0.4 percent. Since the expected design concentration will be in the range of 5 to 7 percent, this cardiotoxicity profile will preclude the use of CF_3I for total-flooding applications in normally occupied areas. However, CF_3I is still a highly promising alternative in total flooding (unoccupied areas) and streaming applications. The potential for carcinogenic effects has not been completely evaluated to date. Although the screening test results are mixed, the available information is insufficient to prevent the safe use of CF_3I in firefighting applications. Further testing (90-day subchronic and full-life chronic bioassay tests) will need to be performed to evaluate preliminarily the carcinógenic potential in whole animal systems.

CF_3I has a strong tendency to decay photolytically in the visible light spectrum, resulting in an atmospheric lifetime of less than 1 day. The most current estimated ODP for CF_3I is 0.0001. Due to the short atmospheric lifetime of the chemical and the photolytic decomposition mechanism, the GWP will be essentially equal to 1.0 for all time horizons, the same as CO_2. Detailed kinetic data and three-dimensional modeling efforts currently in progress are expected to reduce the atmospheric lifetime and ODP values.

There are two declared US manufacturers of CF_3I and other FICs: Pacific Scientific and West Florida Ordnance (WFO). Pacific Scientific is in the process of increasing production from 50 to 500 pounds/day and is building a new plant in Oklahoma that will have an ultimate 3-5 million pounds/yr capacity. WFO has a capacity of 100 pounds/8-hour shift and, depending on demand, could operate multiple production shifts. WFO is in the process of constructing a new plant in Tennessee. There are also several producers of laboratory quantities of fluoroiodocarbon (FIC) chemicals, as well as production in locations such as Russia and Japan. Ultimate price predictions for CF_3I vary widely, from a low of $10 to about $25/pound for annual production quantities of several million pounds.

Pacific Scientific submitted CF_3I under the tradename Triodide® for SNAP listing in May 1994 for total flood application in unoccupied spaces. Triodide® was proposed to be acceptable for unoccupied areas in September 1994. In September 1994, Pacific Scientific also submitted Triodide® for SNAP listing in streaming applications. They are currently awaiting EPA action.

As with Halon 1301, the chemical appears to be compatible with normal materials of construction (stainless steel, carbon steel, brass, and aluminum) to temperatures of at least 77 °C (170 °F). The chemical also appears to be compatible with EPDM, nitrile, and neoprene rubbers to 77 °C (170 °F). The chemical appears to be stable indefinitely in the absence of light, oxygen, and water at temperatures below 116 °C (240 °F).

The toxicological, environmental, and firefighting results to date indicate that CF_3I is a highly promising halon replacement candidate for total-flooding of unoccupied areas and streaming applications. The testing of CF_3I was initiated 18 months ago and has thus far demonstrated that CF_3I meets all the criteria established for an environmentally acceptable drop-in replacement for Halons 1301 and 1211. CF_3I has become a commercial reality. This rapid development is the result of a concerted interdisciplinary effort by a number of personnel and organizations with foresight to obtain an agent that more completely meets the needs of the halon user community.

Consequently, CF_3I is an excellent potential replacement in space critical applications. Note, however, a risk assessment related to design requirements (engineering considerations) and toxicity is required prior to using CF_3I as a fire suppression or inertion agent.

B. RECOMMENDATIONS

The following recommendations are made:

(1) Large-scale testing should be performed based upon user specific applications to confirm the effectiveness results developed to date. Large-scale testing would include that required for UL and FM listings.

(2) Studies of decomposition products resulting from the extinguishing process should be performed.

(3) Nitrogen solubility should be developed for CF_3I.

(4) The thermal operating envelope for CF_3I should be developed.

(5) The hot surface (pyrolysis) products of CF_3I should be determined.

(6) The subchronic and developmental toxicity should be determined. Additional toxicity studies may be required if adverse effects for endpoints are determined in the subchronic test.

(7) CF_3I blends should be investigated to determine whether toxicity considerations could be reduced and extinguishment efficiency maintained.

SECTION X
FUTURE DEVELOPMENT WORK

The program to assess the capabilities and limitations of CF_3I has advanced rapidly in the past 18 months. An interim meeting of the Working Group to assess progress and future requirements was held at NMERI (23 August 1994). The group concluded that research and development efforts were progressing satisfactorily and that in some areas further research would be required to provide a complete picture of CF_3I characteristics. The major areas of concern at this time were the hot surface (pyrolysis) products of CF_3I, its thermal operating envelope, the effects of water vapor and other impurities in storage, and its subchronic and developmental toxicity. Furthermore, it was determined that medium-scale testing should be initiated and that the NRL 2000-ft^3 chamber would be an appropriate test bed for determining the distribution, stratification, and other parameters needed to progress to full-scale testing. Future full-scale testing will be dependent on the applications.

REFERENCES

1. NFPA 2001, *Standard on Clean Agent Fire Extinguishing Systems*, National Fire Protection Association, Quincy, Massachussetts, 1994.

2. US Environmental Protection Agency, "Protection of Stratospheric Ozone," *Federal Register*, Vol. 59, No. 165, pp. 44240-44256, August 26, 1994.

3. DiNenno, P. J., Forssell, E. W., Peatross, M. J., and Wong, J. T., "Thermal Decomposition Testing of Halon Alternatives," *Proceedings of Halon Alternatives Technical Working Conference, 1993*, p. 161, Albuquerque, New Mexico, 1993.

4. Dierdorf, D.S., Moore, T.A., and Skaggs, S.R., "Decomposition Byproduct Analysis During Intermediate-Scale (645 ft^3) and Laboratory Scale (6.18 ft^3) Testing of NFPA 2001 Agents," Proceedings of Halon Alternatives Technical Working Conference, Albuquerque, New Mexico, 10-13 May 1993.

5. Walters, E. A., and Molina, M. J., "Global Environmental Characteristics of Second-Generation Halon Replacements," *Proceedings of Halon Alternatives Technical Working Conference, 1993*, p. 31, Albuquerque, New Mexico, 1993.

6. Skaggs, Stephanie R., "Second-Generation Halon Replacements," *Proceedings of Halon Alternatives Technical Working Conference, 1993*, p. 239, Albuquerque, New Mexico, 1993.

7. Paskovich, D., Gaspar, P., Hammond, G., "Simplified Method for the Preparation of Fluoroalkyl Iodides,"*Journal of Organic Chemistry*, 32, pp. 833-834, 1967.

8. "Fire Extinguishing Agents," Final Report for Contract W44-009-ENGR-507, AD654322, Purdue Research Foundation and Department of Chemistry, Purdue University, with Army Engineers Research and Development Laboratories, Fort Belvoir, Virginia, 1950.

9. Tapscott, R., "Second Generation Chemical Replacements for Halon, "*Proceedings of the First International Conference on Fire Suppression*, pp. 327-335, Stockholm, Sweden, May 1992.

10. Sheinson, R. S., Penner-Hahn, J. E., and Indritz, D., "The Physical and Chemical Action of Fire Suppressants," *Fire and Safety Journal*, Vol. 15, pp. 437-450, 1989.

11. Skaggs, S. R., Heinonen, E. W., Moore, T. A., and Kirst, J. A., *Low Ozone-Depleting Halocarbons as Total-Flood Agents; Volume 2: Laboratory-Scale Fire Suppression and Explosion Prevention Testing*, EPA-6001R-95-150b, August 1995.

12. Moore, T. A., Moore, J. P., and Floden, J. R., "Technology Transfer for New Laboratory Apparatuses for Fire Suppression Testing of Halon Alternatives," Proceedings of the International Conference on CFC and Halon Alternatives, Baltimore, Maryland, 27-29 November 1990. Not paginated.

13. "Cup Burner Test Results," *Center for Global Environmental Technologies Update Series, CGET 93-1*, Center for Global Environmental Technologies, University of New Mexico, Albuquerque, New Mexico, 1994.

14. Scheinson, R., *US Navy CFC-Halon News*, Navy CFC and Halon Clearing House, Vol. 4, No. 3, Washington, DC, September 1994.

15. Lu, G., Johnson, L. L., Ling, M. S., Krantz, J. C., "The Anesthetic Properties of Certain Fluorinated Hydrocarbons and Ethers," *Anesthesia* XLI, 14:467-472, 1953.

16. Mantech Environmental Technologies, *Acute Inhalation Toxicity Study of Iodotrifluoromethane in Rats*, Study No. 1, Project No. 6030-012, August 1993.

17. Mantech Environmental Technologies, *Acute Inhalation Toxicity Study of Iodotrifluoromethane in Rats*, Study No. 2, Project No. 1530-001, March 1994.

18. Haskell Laboratory, *Toxicity Summary for Bromochlorodifluoromethane*, Update, April 11, 1983.

19. Haskell Laboratory, *Toxicity Summary Freon® 11*, Update, October 24, 1988.

20. Kinkead, E. R., Salins, S. A., Wolfe, R. E., Leahy, H. F., English, J. H., *Acute Toxicity Evaluation of Halon Replacement Trifluoroiodomethane (CF_3I)*, Armstrong Laboratory, Human Systems Division, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, January 1994.

21. Deickmann, W. B., and Gerarde, H. W., *Toxicology of Drugs and Chemicals*, Academic Press, New York, New York, 1969.

22. Williams, R. J., Vinegar, A., Byczkowski, J. Z., Creech, J. R., Black, R. K., Neurath, S. K., and Jepson, G. W., *Gas Uptake Kinetics of Bromotrifluoromethane (Halon 1301) And Its Proposed Replacement Iodotrifluoromethane (CF_3I)*, Toxicology Division, Occupational and Environmental Health Directorate, Armstrong Laboratories, Wright-Patterson AFB, Ohio, 1994.

23. Levy, A. G. and Lewis, T., "Irregularities Resulting from the Inhalation of Low Percentages of Chloroform Vapor, and Their Relationship to Ventricular Fibrillation," *Heart*, 3:99-111, 1911-1912.

24. Reinhardt, C. F., Azar, A., Maxfield, M. E., Smith, P. E., Mullen, L. S., "Cardiac Arrhythmias and Aerosol 'Sniffing'," *Archives of Environmental Health*, 22:265-279, 1971.

25. Reinhardt, C. F., Mullin, L. S., Maxfield, M. E., "Epinephrine-induced Cardiac Arrhythmia Potential of Some Common Solvents," *Journal of Occupational Medicine*, 15(12):953-955, 1973.

26. US Environmental Protection Agency, *SNAP Technical Background Document: Risk Screen on the Use of Substitutes for Class I Ozone-depleting Substances, Fire Suppression and Explosion Protection (Halon Substitutes),* U. S. Environmental Protection Agency, Office of Air and Radiation, Stratospheric Protection Division, Washington, DC, March 1994.

27. Beck, P. S., Clark, D. G., and Tinston, D. J., "The Pharmacologic Actions of Bromochlorodifluoromethane (BCF)," *Toxicology and Applied Pharmacology*, 24:20-29, 1973.

28. "Programme for Alternative Fluorocarbon Toxicity (PAFT) Testing," PAFT Management Committee Chairman, % ICI Chemicals and Polymers, Ltd., Cheshire, United Kingdom, April 1993.

29. Meridian Research, Inc., *Assessment of Fire Fighter Exposure to HCFC-123 During Fire Extinguisher Use in Aircraft Hangars,* Submitted to American Pacific Corporation, Las Vegas, Nevada, March 1993.

30. Meridian Research, Inc., *Assessment of Fire Fighter Exposure to HCFC-123 During Extinguishant Efficiency Tests Conducted at the United States Naval Air Station in Beaufort, South Carolina,"* Submitted to Global Change Division, Office of Air and Radiation, Environmental Protection Agency (Washington, DC), and American Pacific Corporation, March 1993.

31. Midwest Research Institute, "Assessment of Occupational Hazards of Firefighter Training," AL/OEMI, Armstrong Laboratory, Brooks AFB, Texas, February 1993.

32. Hodgson, E., Mailman, R. B., and Chambers, J. E., *Dictionary of Toxicology*, Van Nostrand Reinhold Company, New York, p. 243, 1988.

33. HDi, *TOPKAT Toxicity Estimation Report for Mantech Environmental Technology, Inc.*, Rochester, New York, November 1993.

34. Longstaff, E., Robinson, M., Bradbrook, C., Styles, J. A., and Purchase, I. F. H., "Genotoxicity and Carcinogenicity of Fluorocarbons: Assessment by Short-term in Vitro Tests and Chronic Exposure in Rats," *Toxicology and Applied Pharmacology,* 72:15-31, 1981.

35. US Environmental Protection Agency, "Notice of Final Rulemaking, Significant New Alternative Policy," *Federal Register*, 40 CFR Part 82, March 4, 1994.

36. Foltz, V. C. and Fuerst, R., "Mutation Studies with *Drosophila Melanogaster* Exposed to Four Fluorinated Hydrocarbon Gases," *Environmental Research*, 7:275-285, 1974.

37. Garrett, S. and Fuerst, R., "Sex-linked Mutations in *Drosophila* After Exposure to Various Mixtures of Gas Atmosphere," *Environmental Research*, 7:286-293, 1974.

38. Haskell Laboratory, *Toxicity Review Fluorocarbon 152a*, updated June 1, 1987.

39. Seckar, J., Trochimowicz, H. J., and Hogan, G. K., "Toxicological Evaluation of Hydrochlorofluorocarbon 142b," *Journal of Chemical Toxicology*, 24(3):237-240, 1986.

40. Fahr, A., Nayak, A. K., and Huie, R. E., "Ultraviolet Absorption Spectrum of CF_3I," *Proceedings of Halon Options Technical Working Conference, 1994*, p. 465, Albuquerque, New Mexico, May 1994.

41 Solomon, S., Burkholder, J. B., Ravishankara, A. R., and Garcia, R. R., "Ozone Depletion and Global Warming Potentials of CF_3I," *Journal of Geophysical Research*, Vol. 99, pp. 20929-20935, 20 October 1994.

42. US Environmental Protection Agency, *Questions and Answers on Halons and Their Substitutes, Rev. 7*, US Environmental Protection Agency, Office of Air and Radiation, Stratospheric Protection Division, September 1994.

43. ASTM, *Annual Book of ASTM Standards*, 1992 edition, The American Society for Testing and Materials, Philadelphia, Pennsylvania, 1992.

APPENDIX A

LIST OF CF_3I WORKING GROUP ACTIVE MEETING ATTENDEES

NAME	ORGANIZATION/ ADDRESS	PHONE
Mike Bennett	WL/FIVS, Bldg. 63 WPAFB, OH 45433-6553	(513) 255-6302
Martin Blackmore	UK MOD DGFS(ES)/ES 225, Room 34 Block K Foxhill, Bath Avon BA1 5AB England United Kingdom	44 225 884542
Mike Briscoe	Arco Alaska, Inc. 700 G. Street, MS AT01868 Anchorage, AK 99501	(907) 263-4897
David Catchpole	Environment & Regulatory Affairs MB2-1 BP Exploration (Alaska) Inc. 900 East Benson Blvd. P.O. Box 196612 Anchorage, AK 99519-6612	(907) 564-5038
Mike Clauson	Commander TACOM/TARDEC Survivability Technical Center Attn: AMSTA-JS (AMSTA-RSS)(M. Clauson) Warren, MI 48397-5000	(313) 574-5948
Tom Cortina	Halon Alternatives Research Corporation (HARC) 1025 Connecticut Avenue, N.W., Suite 712 Washington, DC 20036	(202) 223-6166
Mark Cummings	U.S. Coast Guard Research and Development Center Avery Point Groton, CT 06340-6096	(203) 441-2761
Doug Dierdorf	CGET/NMERI The University of New Mexico 901 University Blvd. S.E. Albuquerque, NM 87106-4339	(505) 272-7256
Jerry Everard	Alyeska Pipeline Service Company Mailstop 559 1835 South Bragaw Street Anchorage, AK 90512	(907) 265-8165
Rich Hansen	U.S. Coast Guard Research and Development Center Avery Point Groton, CT 06340-6096	(203) 441-2866
Craig Herud	USA CSTA SPECS-EN-PC, Building 370 Aberdeen Proving Ground, MD 21005	(301) 278-3165
Larry Hilton	Deepwater Iodides P.O. Box 17599 Irvine, CA 92713	(800) 854-4064
Frank Hughes	West Florida Ordnance Inc. Suite 111 41N. Jefferson St. Pensacola, FL 52501	(904) 433-2221
John Hughes	CF_3I Defense Procurements 711 Bennaville Street Birmingham, MI 48009	(313) 646-6445

CF3I MEETING ATTENDEE LIST

NAME	ORGANIZATION/ ADDRESS	PHONE
Major Gary Jepson	AL/OET WPAFB, OH	(513) 255-8936
Charles Kibert	Air Base Fire Protection and Crash Rescue Systems Branch WL/FIVCF 139 Barnes Drive Tyndall AFB, FL 32403-5319	(904) 283-3734
Joe Macko	U.S. Army Materiel Command Attn: AMCRD-E 5001 Eisenhower Avenue Alexandria, VA 22333	(703) 274-0815
Alexander Maranghides	Navy Technology Center for Safety & Survivability Code 6180 Naval Research Laboratory Washington, DC 20375-5000	(202) 404-8101
Bill Meserve	Pacific Scientific HTL/Kin Tech Division 1800 Highland Avenue Duarte, CA 91010	(818) 359-9317
Ted Moore	CGET/NMERI The University of New Mexico 901 University Blvd. S.E. Albuquerque, NM 87106-4339	(505) 272-7261
Larry Motschenbacher	Alyeska Pipeline Service Company Mailstop 559 1835 South Bragaw Street Anchorage, AK 90512	(907) 265-8165
Steve Newhouse	Pacific Scientific Corporation 1800 Highland Avenue Duarte, CA 91010	(818) 359-9317 Ext. 275
Jon Nimitz	The IKON Corp. 3300 Mountain Road N.E. Albuquerque, NM 87106	(505) 256-1463
Adam T. Richardson	Control Fire Systems, Inc. 97 Six Point Road Toronto, Ontario M8Z 2X3 CANADA	(416) 236-2372
Neil Richardson	Pacific Scientific Corporation 1800 Highland Avenue Duarte, CA 91010	(818) 359-9317
Jack Riley	JR & Associates (Halotron) 1815 15th Avenue Menominee, MI 49858	(906) 863-8509
R. K. Schlilaty	The Boeing Company	(206) 234-9939
Stephanie R. Skaggs	CGET/NMERI The University of New Mexico 901 University Blvd. S.E. Albuquerque, NM 87106-4339	(505) 272-7262

CF3I MEETING ATTENDEE LIST

NAME	ORGANIZATION/ ADDRESS	PHONE
Robert E. Tapscott	CGET/NMERI The University of New Mexico 901 University Blvd. S.E.. Albuquerque, NM 87106-4339	(505) 272-7252
Marco Tedeschi	Naval Air Warfare Center Aircraft Division Code SR11 Lakehurst, NJ 08733-5100	(908) 323-1107
Dennis Zallen	Zallen International Associates (ZIA) 14216 Turner Court N.E. Albuquerque, NM 87123	(505) 299-3025

CF_3I Meeting Attendees
8/23/94

Maria Elena Arner Geo-Centers, Inc. 1755 Jefferson Davis Hwy. Suite 910 Arlington, VA 22202 Phone: 703-769-1899 Fax: 703-769-1885	
Dr. Dana Brabson New Mexico Engineering Research Institute The University of New Mexico Chemistry Department Albuquerque, NM 87131 Phone: 505-277-1649 Fax: 505-277-2609	
Mr. Mike Briscoe Prudhoe Bay Projects and Construction Arco Alaska, Inc. P.O. Box 100360 Anchorage, AK 99510-0360 Phone: 907-263-4897 Fax: 907-265-1555	
Mr. David Catchpole BP (Exploration) Alaska Environment & Regulatory Affairs MB2-1 P.O. Box 196612 900 East Benson Blvd. Anchorage, AK 99519-6612 Phone: 907-564-5038 Fax: 907-564-5020	
Dr. Douglas S. Dierdorf Pacific Scientific Corp. 3916 Juan Tabo N.E., Suite 12 Albuquerque, NM 87111 Phone: 291-1109 Fax: 291-1141	
Mr. William Elliot Hughes Aircraft Compapny Santa Barbara Research Center 75 Coromar Drive Goleta, CA 93117 Phone: Fax:	

Dr. George C. Harrison Walter Kidde Aerospace, Inc. 4200 Airport Drive N.W. Wilson, NC 27893 Phone: 919-237-7004 Fax: 919-237-4717	
Major Gary Jepson AL/OET Bldg. 79, Area B Wright-Patterson AFB, OH 45433-7400 Phone: 513-255-8936 Fax: 513-255-1474	
Charles J. Kibert WL/FIVCF Stop 39 139 Barnes Drive, Ste. 2 Tyndall AFB, FL 32403-5323 Phone: 904-283-3734 Fax: 904-283-9797	
Matt Kolleck Booz-Allen Hamilton 4141 Col. Glenn Hwy, Suite 131 Dayton, OH 45431 Phone: 513-429-9509 Fax: 513-429-9795	
Mr. Joe Macko U.S. Army Materiel Command ATTN: AMCRD-E 5001 Eisenhower Ave. Alexandria, VA 22333 Phone: 703-274-0816 Fax: 703-274-5146	
Alexander Maranghides Geo-Centers Inc. 10903 Indian Head Highway, Ste. 502 Fort Washington, MD 20744 Phone: 202-967-2766 Fax: 202-767-1716	
Mr. Bob McDonald ARCO Exploration and Production 2300 West Plano Parkway Dallas, TX 75075-8499 Phone: 214-754-3583 Fax: 214-754-3069	

Ted A. Moore New Mexico Engineering Research Institute The University of New Mexico 901 University Blvd. S.E. Albuquerque, NM 87106-4339 Phone: 505-272-7261 Fax: 505-272-7203	
Mr. Steve Newhouse Pacific Scientific Corp. 1800 Highland Avenue Duarte, CA 91010 Phone: 818-359-9317, ext. 275 Fax: 818-358-5609	
Dr. Jon Nimitz The IKON Corporation 3300 Mountain Road N.E. Albuquerque, NM 87106-1920 Phone: 505-256-1463 Fax: 505-256-1003	
Stephanie R. Skaggs New Mexico Engineering Research Institute The University of New Mexico 901 University Blvd. S.E. Albuquerque, NM 87106-4339 Phone: 505-272-7262 Fax: 505-272-7203	
Robert E. Tapscott New Mexico Engineering Research Institute The University of New Mexico 901 University Blvd. S.E. Albuquerque, NM 87106-4339 Phone: 505-272-7252 Fax: 505-272-7203	
Bob Tetla WL/FIVCF, Stop 39 139 Barnes Drive, Ste. 2 Tyndall AFB, FL 32403-5323 Phone: 904-283-3734 Fax: 904-283-9797	
Allen Vinegar ManTech Environmental Technology P.O. Box 31009 Dayton, OH 45437 Phone: 513-256-3600 Fax: 513-258-2197	

APPENDIX B

CF_3I NEWSBRIEFS

CF_3I NEWSBRIEF *10 AUG 93*

1.0 TACOM Test Program

Testing was performed on two potential Halon 1301 replacement agents at New Mexico Engineering Research Institute (NMERI) August 10-12 on behalf of TACOM. Testing was performed on both "Halon 13001" (CF_3I) and PGA (dry chemical) to determine suppression capabilities in extinguishing tank engine fires. No full-scale testing of Halon 13001 had previously been conducted.

Protocol:

A mock-up of a tank compartment from the M-1 Abrams main battle tank was built at the NMERI testing facility. The compartment consisted of a circular JP-4 fuel pan, a circular tube located above the pan, a spray nozzle to spray JP-4 into the compartment, and two parallel lines on each side of the cylindrical tube used to discharge the agent into the compartment.

For baseline precision and accuracy, all tests began with the ignition of the fuel pan. Pre-burn continued until the compartment temperature was allowed to reach 400°C, at which time the fuel spray was started. Ten seconds after the fuel spray was initiated, the agent was discharged into the compartment. If the fire was suppressed, the fuel spray then continued for 30 seconds to simulate real-time reignition potential. The test was considered successful if the fire was both extinguished and prevented from reigniting. If the fire was not extinguished or if the fuel reignited, the failed suppression attempt was extinguished using Halon 1211.

Table I NMERI Testing, 10 AUG 93

Test #	Agent	Wt of Agent (lbs)	Pressure (psi)	Fire Suppressed
1	Halon 1301	3	750	Yes
2	n/a	n/a	n/a	n/a
3	Halon 13001	4	800	No*
4	Halon 13001	6	750	Yes
5	Halon 13001	4.4375	610	Yes

Results:

Preliminary results illustrated in Table I on the previous page and Table II below indicate that CF_3I is at least as effective as CF_3Br under controlled conditions. However, due to the limited availability of CF_3I, more laboratory scale testing must be conducted to validate these results and to determine if CF_3I is actually physically and chemically comparable to CF_3Br as a potential drop-in replacement. The results of the tests are provided below. Halon 1301 and Halon 13001 tests were conducted using pressurized N_2 in a 204 in^2 cylinder. Various pressures and weights of Halon 13001 were evaluated.

Table II *NMERI Testing, 11 AUG 93*

Test #	*Agent*	*Wt of Agent (lbs)*	*Pressure (psi)*	*Fire Suppressed*
1	*Halon 13001*	*2.816*	*560*	*Yes*
2	*Halon 1301*	*3*	*750*	*Yes*
3	*Halon 1301*	*2.5*	*500*	*No*

Opinion:

During the initial Halon 13001 testing [*] it appeared the fire was blown out of the tank compartment, possibly to the excessive pressure which allowed the fire burn unsuppressed. Preliminary analysis indicates that lower pressure may be required for Halon 13001 than for Halon 1301 due to slight variations in field densities (the density 13001 is greater than that of 1301). Further research needs to be accomplished to verify this.

Conclusion:

The use of Halon 13001 as a replacement for Halon 1301 appears promising. However, more research must be accomplished to look into the various properties and the feasibility of using Halon 13001 as a replacement to Halon 1301. Further research may include; toxicity, global warming potential, ozone depletion potential, decomposition, nitrogen solubility, etc. Results of NMERI testing demonstrates that Halon 13001 is as efficient and possibly more efficient on at least a volumetric basis than Halon 1301.

2.0 General Testing Program At NMERI

Funding

19 August, the first increment of funding (North Slope Oil and Gas Producers, $25K) became available to initiate the program. This funding will be utilized to acquire the materials necessary to initiate Task 2 and begin activities required on all Tasks.

Results to Date

Task 1: Agent Synthesis

Subtask 1.1 Agent Production

Quantities of CF_3I in 10 to 12 kg lots are being acquired by the HTL/Kin-Tech Division of Pacific Scientific Company (Pac-Sci). This firm is using their fast, flexible purchasing ability to procure usable quantities of agent, and are redistributing this agent in support of testing programs at NMERI, US Army-TACOM, NIST, US Air Force. Armstrong Laboratory, and U.S. Air Force-Wright Laboratories. At least two manufacturing organizations, one associated with Pac-Sci and another independently represented by Mr. John Hughes, appear to be ready to deliver developmental quantities in the next four to eight weeks.

Contacts

Mr. Steve Newhouse, Pacific Scientific
(818) 359-9317
Mr. John Hughes (independent representative of an unnamed chemical concern)
(313) 646-6445.

Subtask 1.2 Cost Assessment

To be initiated promptly

Subtask 1.3 Agent Solubility

Deferred at the request of ARCO Alaska, Inc. (Letter dated 28 Jun 93)

Task 2: Stability/Material Compatibility

Subtasks 2.1 and 2.2

The start of this program has been due to late funding. Test preparation is being started immediately, the six month test period will begin on or before 1 Oct 93.

Task 3: Toxicology

Subtask 3.1 Limit Testing

Iodotrifluoromethane was administered as a vapor for fifteen minutes to one group of five male and five female Sprague Dawley rats on 8/13/93. The chamber concentration was adjusted based on real-time analysis using an IR analyzer. Due to the high desired chamber concentration (60000ppm), the chamber samples had to be diluted for analysis. An error in determining the actual concentration in the chamber resulted in an exposure concentration approximately twice (127289ppm) that desired. This 12.7% concentration exceeded the required extingushment concentration by a factor of four. Although all rats demonstrated severe salivation, no rats died and all recovered within one-hour post exposure in addition to retaining normal appearance for 14 days.

Subtask 3.2 LC_{50}

Agent was shipped to Armstrong Laboratory Week 26 July and tests are progressing on the initial schedule.

Subtask 3.3 Decomposition Products

To be initiated as soon as practical at Armstrong Laboratory.

Task 4: Fire Extinguishment/Inertion Tests

Subtask 4.1 Cup Burner Tests

To be performed Oct 93.

Subtask 4.2 Small Scale Tests
Two tests were performed at a concentration of 3.4 vol% (110% of Cup Burner). Performance was similar or slightly superior to Halon 1301 at the same concentration.
Subtask 4.3 Inertion Tests
Explosion sphere work scheduled in the October - November time Period.

Note: Large scale tests were conducted in the NMERI MI Engine Compartment Simulator on 10-11 Aug 93. In this test fixture, 3 lb of Halon 1301 consistently extinguish the test fire and 2.5 lb. consistently fails. The fire was successfully extinguished with 2.8 lb of CF_3I. Halon controls were performed immediately after the test. These controls exhibited normal results.

Task 5: Global Environmental Impacts
Work has begun at several locations (UNM, NIST, LLNL, Army) to verify the predicted zero ODP/GWP values for CF_3I.

While work on major portions of this program have been delayed by late funding, work on other areas is progressing well. The delay in initiation of Task 2 will unavoidably delay the completion of the program by two months. I suggest that the mid-point meeting be delayed to early December so that preliminary (45 day) stability and compatibility data will be available.

CF_3I INERTION SPHERE TESTING

by Everett Heinonen and Doug Dierdorf

Center for Global Environmental Technologies
University of New Mexico
901 University Blvd. SE, Albuquerque, NM 87106
(505) 272-7250

Background

One of the largest uses of Halon 1301 is for prevention of explosions in environments containing combustible gases or dusts. The application of Halon 1301 for explosion prevention is a critical requirement in Alaskan North Slope petroleum production and handling facilities.[1]

For many years, the standard method of evaluating the effectiveness of an inerting agent has been through the use of the inertion sphere.[2] The NMERI Inertion Sphere has been used extensively to evaluate the performance of halon replacements.[3,4] Results in the NMERI sphere compare well with the work of other experimenters,[2] especially when propane is used as the fuel.[5] A complete evaluation of the flammability envelope for the system of trifluoroiodomethane (CF_3I) is required to qualify CF_3I for further development as an explosion prevention agent.

Experimental

An extensive series of test were performed in the NMERI Inertion Sphere to determine the shape and limits of the flammability envelope for the system CF_3I-propane-air. The detailed experimental procedure has been reported previously.[3] Thirty-four distinct mixtures of CF_3I, propane and air were tested in the inertion sphere. An ignition spark was applied to each mixture, and the pressure increase in the sphere was measured. A pressure increase of 1 psi or greater was considered to be an explosion. The ignition spark was formed between two centrally located electrodes separated by 6 mm by discharge of a capacitor bank charged to 70 Joules. Figure 1 contains a plot of the CF_3I results; Halon 1301 data[4] are shown for comparison. Table 1 contains the composition and pressure increase data.

Conclusions

Based on the data obtained in this test series, the flammability envelopes for CF_3I and Halon 1301 are essentially identical. This result implies a storage volume equivalent of approximately 0.9 and a weight equivalent of 1.3 for CF_3I in explosion prevention applications.

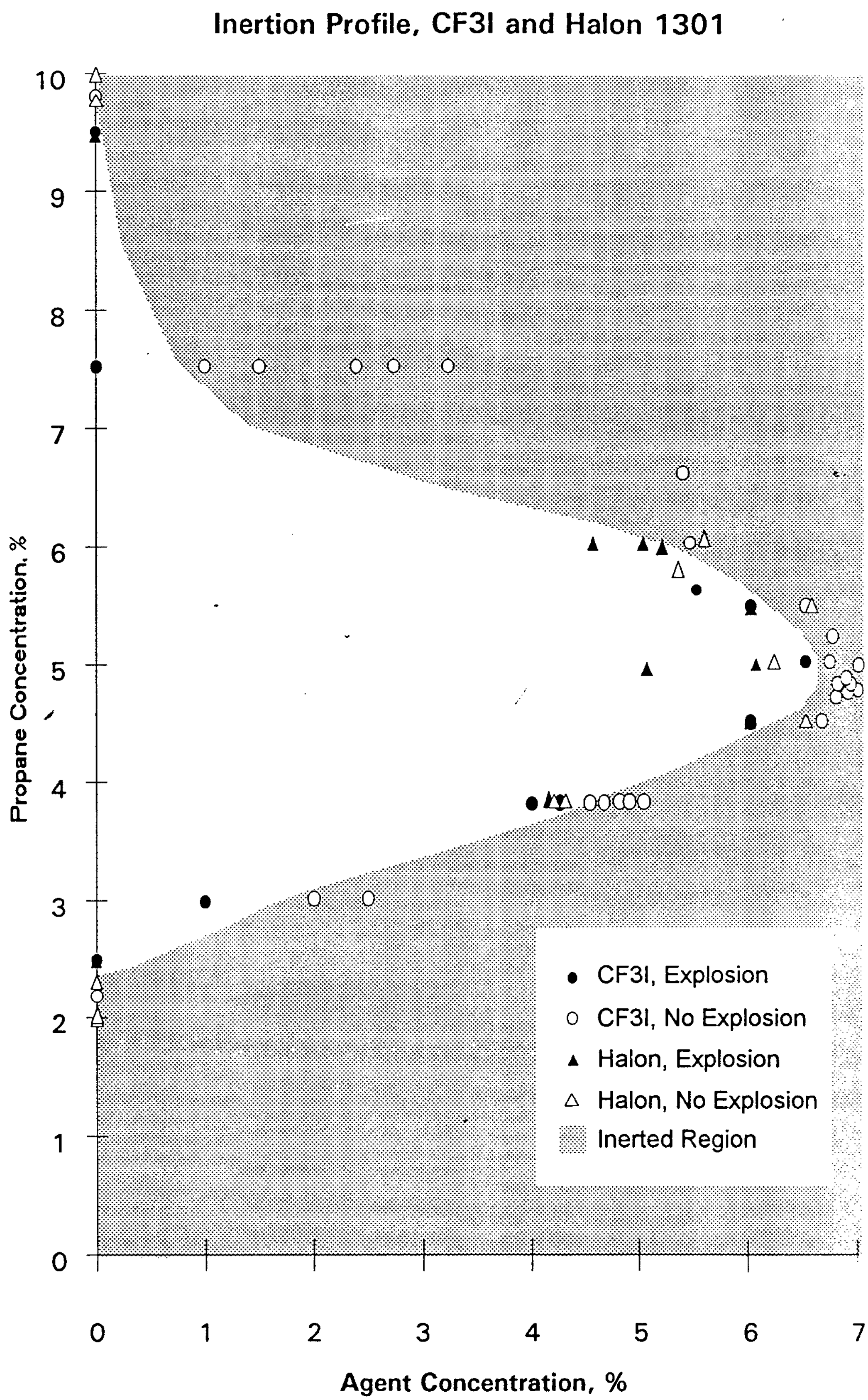


Figure 1. Flammability Envelop for the system CF_3I, propane, and air.

Table 1. CF_3I Inertion Sphere Data

Propane, %	CF3I, %	Pressure Increase, psi	Propane, %	CF3I, %	Pressure Increase, psi
7.5	3.2	0.0	4.8	6.8	0.0
7.5	2.7	0.0	4.8	7.0	0.0
7.5	2.4	0.0	4.8	6.9	0.0
7.5	1.5	0.0	4.7	6.8	0.0
7.5	1.0	0.0	4.5	6.7	0.0
7.5	0.0	36.0	4.5	6.0	43.7
6.6	5.4	0.0	4.5	6.0	43.7
6.0	5.4	0.0	3.8	5.0	0.0
5.6	5.5	1.1	3.8	4.9	0.0
5.5	6.5	0.2	3.8	4.8	0.0
5.5	6.0	1.0	3.8	4.7	0.0
5.2	6.8	0.0	3.8	4.5	0.0
5.0	7.0	0.0	3.8	4.3	62.9
5.0	6.7	0.6	3.8	4.0	68.0
5.0	6.5	27.0	3.0	2.5	0.0
4.9	6.9	0.1	3.0	2.0	0.0
4.8	6.9	0.0	3.0	1.0	76.6

1. Pope, P. R., "North Slope Oil field Fire Protection: the Halon Issue," *Proceedings of the International Conference on CFC and Halon Alternatives*, Washington, DC (1989).

2. Dazell, W. G., *A Determination of the Flammability Envelope of Four Ternary Fuel-Air-Halon 1301 Systems*, Fenwal Report No. PSR-624, Fenwal Incorporated, Ashland MA (1977).

3. Heinonen, E. W., and Skaggs, S. R., "Fire Suppression and Inertion Testing of Halon 1301 Replacement Agents," *Proceedings Halon Alternatives Technical Working Conference 1992,* Albuquerque, NM (1992).

4. Heinonen, E. W., Skaggs, S. R., and Kirst, J. A., *Perfluorocarbons as Total-Flood Extinguishing Agents Task 3 - Inertion and Fire Suppression Testing at Laboratory Scale*, NMERI OC 92/26, The New Mexico Engineering Research Institute, The University of New Mexico, Albuquerque, NM (1992).

5. Heinonen, E. W., "Effects of Ignition Source and Strength on Sphere Inertion Results," *Proceedings Halon Alternatives Technical Working Conference 1993,* Albuquerque, NM (1993).

CF_3I NEWSBRIEF No.2 — 12 OCT 93

Air Base Crash Rescue and Fire Protection Section (WL/FIVCF)
Tyndall AFB, Florida 32403 Tel: (904) 283-3745 Fax: (904) 286-6763

1. INDUSTRY COMMITMENT:

Pacific Scientific has announced their corporate commitment to the commercialization of CF_3I as a Halon 1301 replacement. Based on the positive outcome of testing, they are in the process of forming industrial partnerships that will insure an adequate supply of iodine for synthesis of the agent. Steve Newhouse, product line director for Pacific Scientific, has stated that 1,000 to 2,000 pounds will be available by the end of the year for use in testing. He badly needs information from all interested parties on their requirements for CF_3I for the next year. Please send your requirements to Steve Newhouse at Pac Sci:

Tel:818-359-9317 Fax: 818-359-7013

or Charles Kibert at Tyndall AFB. Steve is looking into a near term 10,000 pound run and if we can assure its purchase the agent will be available for approximately $100 a pound.

2. EPA SNAP:

As a function of Pac Sci's commitment to CF_3I production and systems, they will take on the responsibility for submitting the requisite data for EPA SNAP approvals. In a recent meeting with EPA, Pac Sci was informed that the only toxicology test required for approval for unoccupied space use (engine nacelles and dry bays) is an LC_{50} test. This is a limited approval. Cardiac sensitization data is required for approval for occupied space use. Pac Sci is in the process of contracting for the LC_{50} test and we can expect results by mid-December. This is clearly a crucial step in the overall scheme of things, but it does not appear to be a major obstacle.

3. AIRCRAFT APPLICATIONS:

In a recent meeting of the Technology Transition Team (T2 Team) at Wright-Patterson Air Force Base held on 5-7 October 1993 and chaired by Major Sam Carbaugh, CF_3I was selected as one of the final candidates for both engine nacelle and dry bay applications. This was a major milestone for their program, involving the down selection of 3 agents for advanced testing from an original panel of 12 agents. CF_3I was considered only within the last 2 months and as a result of information provided from a number of sources, it was allowed to contend for these Halon 1301 replacement applications. The potential benefits of CF_3I allowed the T2 Team to recommend it for the final test program, in spite of it not yet being a production chemical. Pac Sci's efforts to produce CF_3I were a major factor in allowing its consideration.

4. ODP and GWP:

a. As a consequence of its contract with Major Carbaugh's organization, NIST has arranged with Lawrence Livermore Laboratory (LLL) to perform atmospheric modeling of CF_3I relative to ozone depletion and global warming. Marc Nyden of NIST made a presentation that indicated that CF_3I could cause ozone depletion only if released into the stratosphere. Tropospheric lifetime is on the order of a few days and if released in the troposphere or lower there should not be any transport to the stratosphere and consequent ozone depletion. NIST will provide information on LLL's study when available and this will be a key factor in determining CF_3I's future.

b. Further conversations with EPA indicated that for a complete judgement to be made on this issue, realistic data on aircraft Halon 1301 releases versus altitude should be gathered. Major Carbaugh directed SURVIAC, one of his subs, to gather the data for Air Force aircraft. Similar data should be gathered by the Navy and the FAA. Please provide any information you have to Charles Kibert and he will insure it is forwarded to EPA, NIST, and Pac Sci. It would be worthwhile to have one organization (Air Force, Navy, or FAA serve as a focal point for gathering this information. Please let Charles Kibert know if your organization can take on this task.

5. TOXICITY TESTING:

a. In the last newsbrief it was noted that the Limit Test showed no lethality during a 15 minute exposure in rats at 12.7% CF_3I concentration, approximately 4 times its extinguishing concentration. Capt Gary Jepson presented preliminary data on testing conducted by Armstrong Lab at Wright-Patterson at the T2 Team meeting. He stated that it was very difficult to obtain uptake and absorption of the chemical and that as a consequence there was little or no interaction occurring with animal tissues. This is very good news as it means that the probability of negative toxic effects is greatly reduced.

b. As noted earlier, Pac Sci is contracting for an LC_{50} test. The next testing hurdle is to perform a cardiac sensitization test that will enable us to state whether CF_3I is usable in occupied spaces. This should be no problem as long as it can be demonstrated that LOAEL occurs at greater than the 3.7% concentration (extinguishment + 20%). Capt Jepson has indicated that Armstrong Laboratory will arrange for this testing if: (1) we provide him with 30 Kg of agent, and (2) $50,000. Both issues are being attended to and we hope to have this testing initiated shortly. It should be noted that neither the LC_{50} nor the cardiac sensitization tests were forecast as part of the Phase 1 program agreed to by participants at our NMERI meeting. However it seems that events are beginning to overrun our planning.

c. Other CF_3I toxicology testing is ongoing at Armstrong Labs. The gas uptake studies are continuing. Mutagenic testing will commence shortly. Partition coefficients are being determined and a physiologically based model is being developed. QSAR comparisons between Halon 1301 and CF_3I are being made. A series of combustion toxicology studies is also planned.

6. INERTION TESTING:

A copy of NMERI's inertion testing results was recently distributed to Ad Hoc Committee members by Doug Dierdorf. In case you did not receive it and desire a copy, please contact Doug at NMERI. The data suggests that CF_3I is about equivalent to Halon 1301 in its inertion performance.

7. CORROSION TESTING:

As part of its work presented to the T2 Team, NIST provided some materials compatibility information on CF_3I that stated:

AM355 - intergranular cracking
CDA172 - green residue

AM355 is a common stainless steel sometimes used for agent containers while CDA172 is a copper-beryllium alloy. Copper is envisioned as a scavenger for free iodine during CF_3I synthesis and the presence of a green residue is suggestive of copper iodide, not an unexpected or unusual occurrence. The testing conducted by NIST was at 150°C (302°F) for 30 days. It was noted by the T2 meetin participants that this was a rather severe test and that a more appropriate test should have been conducted at 160°F for the 30 day duration. NMERI is also conducting corrosion testing and we will be sure to scrutinize both their testing and NIST's results to appropriately assess the data. NMERI's test data will be at 30, 60, and 180 days at 170°F.

8. SUMMARY:

a. It is clear that there is every reason to be cautiously optimistic regarding CF_3I's future as a Halon 1301 replacement. Pac Sci's entry into the marketplace with CF_3I will dramatically accelerate the pace of research, development, and testing. A very optimistic scenario puts it into some applications within 1 year, assuming it survives all testing and is shown to function as a drop-in Halon 1301 replacement.

b. The major obstacle at present is the ODP modeling results expected from LLL in the near future, the gathering of aircraft Halon 1301 release data, and EPA's evaluation of both pieces of information. All parties involved in this phase of deliberation are urged to cooperate and freely exchange data.

c. Toxicology testing is showing CF_3I to behave similar to Halon 1301 and the toxicologists are not expecting any surprises.

d. Corrosion and materials compatibility testing are also of vital importance due to the preliminary NIST data. Again this was an unusually severe set of tests and NMERI's results should clarify the situation.

e. NIST will be conducting more extensive CF_3I testing in connection with the Air Force aircraft survivability program. This is a welcome turn of events as it means that much of the data needed for a complete picture of CF_3I will be determined by NIST.

Charles J. Kibert
WL/FIVCF (Stop 37)
139 Barnes Drive, Suite 2
Tyndall AFB, FL 32403-5323

Tel: (904) 283-3745/3746 Fax: (904) 286-6763 DSN: 523-3745/3746

CF_3I NEWSBRIEF No. 3 — 9 February 1994

Air Base Fire Protection and Crash Rescue Section (WL/FIVCF) Editor: Charles Kibert
Tyndall AFB, Florida 32403-5323 Tel: (904) 283-3734 Fax: (904) 286-6763

1. STABILITY TESTS:

The 30 day and 60 day stability test results conducted by NMERI showed no breakdown of the agent in the various configurations: neat, w/ N_2 (300 psi and 600 psi), w/air (300 psi), and with 0.5 % moisture. Tests were conducted at 80°F, 125°F, and 170°F. This is key information that was initially designed to serve as a go/no-go criterion for further CF_3I testing. Stability testing will continue for a full 180 day time frame.

2. MATERIALS COMPATIBILITY TESTS:

Compatibility tests of CF_3I with a variety of materials is ongoing and results are expected in the near future.

3. CREW COMPARTMENT TESTS:

The Army conducted an armored vehicle crew compartment test at Aberdeen Proving Grounds on 3 February 1994 that, as a secondary effort, compared the performance of CF_3I with Halon 1301. The primary objective was to test new detection systems for armored vehicles and only a minimal amount of information was collected relative to agent performance. A 3.5 in. shaped charge was fired through a 1.25 in. thick aluminum plate, simulating a Bradley fighting vehicle's exterior structure. A simulated fuel cell with 13 gallons of JP-8 heated to 155°F was ignited by the charge, creating a fire ball within the crew space. Two 7 lb. charges of agent were discharged in an attempt to suppress the explosion in less than 250 milliseconds. Preliminary indications are that the extinguishment was successful. Combustion gas samples were extracted and will be analyzed for their toxic content. Additional information will be available once the data is analyzed.

4. TOXICITY TESTING:

LC_{50} testing and cardiotoxicity testing are underway. LC_{50} results will be available in April and the cardiotox data in July. All indications thusfar are that CF_3I is very similar to Halon 1301 in its toxicological behavior. Our understanding is that the EPA would issue a tentative ruling that CF_3I is acceptable for general use if the LC_{50} and cardiotox data is positive. For complete EPA acceptance developmental/subchronic tox testing will be required at an estimated cost of $120,000, requiring about 6000 lb. of agent. This last phase of testing should be considered by the Working Group for immediate funding and execution as all information to-date on CF_3I is positive. We can discuss this matter at the next meeting of the Working Group (see last item below).

5. FLUOROIODOCARBON STREAMING AGENTS:

Testing of the first FIC streaming agent candidate, C_3F_7I, is underway with toxicological Limit Tests being conducted by Mantec under contract to NMERI and the Air Force and other Phase 1 tox testing being accomplished at Armstrong Labs (Wright-Patterson AFB, Ohio). Initial fire suppression effectiveness and flow behavior will be conducted at NMERI and Tyndall AFB. As is the case with CF_3I compared to Halon 1301, C_3F_7I has laboratory fire suppression performance similar to Halon 1211. This compares to current generation offerings with only one-half or less the performance of Halon 1211.

6. AGENT COST:

Currently CF_3I is being quoted as low as $50 per pound for large quantities (1000 lb range). At this moment Pacific Scientific is the only manufacturer committed to synthesizing large quantity lots.

7. GLOBAL ENVIRONMENTAL IMPACTS:

The Subcommittee chaired by Marc Nyden at NIST is currently conducting a fax (and facts!) debate as to the data required to fully assess the ODP of CF_3I. Susan Solomon, NOAA, has estimated the atmospheric lifetime at 2 days with the net result that for ground level releases the ODP is essentially zero. Charles Kibert has directed a request to EPA that they publicly or in an appropriate ruling state that CF_3I has acceptable, essentially zero ODP, for ground level applications. Karen Metchis (EPA) made a verbal commitment that this would be an acceptable approach. The data gathering effort for aircraft releases of Halon 1301 is still underway and will be utilized to determine if there are potential problems for aircraft applications. Uncertainties about kinetic rates for several atmospheric reactions may need to be resolved. However, the lack of complete information should not be an obstacle to permitting ground level uses which include, in addition to fire suppression, refrigerants, foam blowing agents, and others. A complete determination of kinetic rates has an estimated cost of $125,000. The actual funding of this work will depend on the aircraft release data. If, as the preliminary information indicates, the vast bulk of releases are below 20,000 ft., then the ODP remains essentially negligible. If however there are significant releases that could migrate to the stratosphere AND the aircraft community determines that CF_3I is still a viable agent for their applications, then this work will have to be accomplished.

7. AD HOC COMMITTEE MEETING:

Per request of Mike Briscoe (ARCO Alaska) and others, a meeting of the Working Group will be held at NMERI on 10-11 March 1994. We will assume that the meeting will begin in the NMERI Conference Room at 9:00 am. The main subject will be the conduct of large scale testing of CF_3I by the Coast Guard on one of their test vessels at Mobile, Alabama. ARCO is intending to participate and share costs with the CG for the 2 tons of agent required at a cost of $50/lb. They would like to discuss the overall situation with CF_3I as well as to obtain input from the Working Group on the proposed program. Please plan to attend. By the way, if you received a copy of this letter you may consider yourself a member of the Working Group. Another agenda item is to discuss Phase 2 work and funding. A further item will be to discuss the need for to fully develop thermodynamic and flow information for use in designing CF_3I systems.

CF_3I NEWSBRIEF No. 4 15 June 1994

Air Base Fire Protection and Crash Rescue Section (WL/FIVCF) Editor: Charles Kibert
Tyndall AFB, Florida 32403-5323 Tel: (904) 283-3734 Fax: (904) 283-9797

1. Document from NMERI Meeting

Accompanying this newsletter is a document that was mentioned at the last meeting of the CF_3I Ad Hoc Working Group Meeting during the Halon Technical Options Conference, 3-5 May 1994 at NMERI. It contains the information that Ron Sheinson presented comparing CF_3I to Halon 1301 extinguishing performance in the CBD 2000 ft^3 chamber. Dr. Sheinson would like to acknowledge the assistance of Pacific Scientific who provided CF_3I for the testing as well as Florida Ordnance who provided CF_3I during the calibration phase of the testing.

2. Release vs Altitude Information

One of the key elements of information that was sorely needed to consider allowing CF_3I in aircraft fire/explosion protection applications was historical release vs. altitude data for Halon 1301. Due to the photolytic decomposition of CF_3I in the visible light spectrum, the ODP of CF_3I is a variable that is largely a function of altitude of release. Through the efforts of Major Carbaugh and Mike Bennett at Wright-Patterson AFB, this data has been compiled. A brief outline is given below. The data is a compilation of U.S. Air Force and U.S. Navy data for the military and Boeing Aircraft Company data for commercial aircraft. The full report will be released to the Working Group when available in its final form. The 9.1 Km altitude equates to the cruising altitude of a military cargo aircraft. The stratospheric ozone layer begins just above this elevation, approximately 10-11 Km.

Annual Agent Halon 1301 Release, Kg				
Altitude	**Military Fires**	**Military False Alarms**	**Commercial (include. cargo bays)**	**Total**
Above 9.1 Km	2.1	2.0	158.2	162.3
Below 9.1 Km	54.0	154.2	775.0	983.2
TOTAL	**56.1**	**156.2**	**993.2**	**1145.5**

The releases in commercial cargo bays clearly constitute the bulk of Halon 1301 releases. It is not known how much fire suppressant actually escapes the cargo hold into the atmosphere versus how much is brought back to ground level when the aircraft lands.

The initial reaction of the U.S. EPA to this data is that these releases should not be a significant factor in the decision to allow the use of CF_3I in aircraft fire suppression applications.

4. Testing Activities

The Air Force has initiated testing of CF_3I and other candidates in dry bay and engine nacelle applications at Wright-Patterson AFB, Ohio. Mike Bennett reports that the first tests are showing CF3I to be significantly more effective than other agents in the dry bay tests, perhaps even more effective than Halon 1301. Engine nacelle tests are just beginning and results should be emerging shortly.

5. Rate Constants

Dr Robert Huie at NIST reported that a Russian group headed by Sergei N. Buben of the Russian Academy of Sciences, working with NIST on the determination of key reaction rates needed to determine the ODP of CF_3I, has begun to produce their first results. The reaction rate for the $IO + O_3$ reaction is estimated at 5×10^{-17} cc/molecule-sec at stratospheric temperatures. According to Dr. Huie this rate is very low and could be considered insignificant. In comparison, a 10^{-10} reaction rate would be considered a fast rate. When the unknown rate constants are determined by NIST and their Russian counterparts, they will be fed into a 3-D model created by Don Wuebbles at LLL to determine what will probably be the "official" ODP for CF3I.

6. Next Meeting

The next meeting of the CF_3I Ad Hoc Working Group will be during the International CFC/Halon Conference in Washington in late October. The exact time and location will be announced during the Conference.

Intermediate Scale CF_3I Testing at Naval Research Laboratory (NRL) Chesapeake Bay Detachment (CBD)

Preliminary Testing and Proposed Test Matrix

Dr. Ronald S. Sheinson, Naval Research Laboratory, Washington, D.C.
CF3I Ad-Hoc Working Group Meeting, Albuquerque, May 3-5, 1994

CF_3I Intermediate Scale Testing @ NRL/CBD

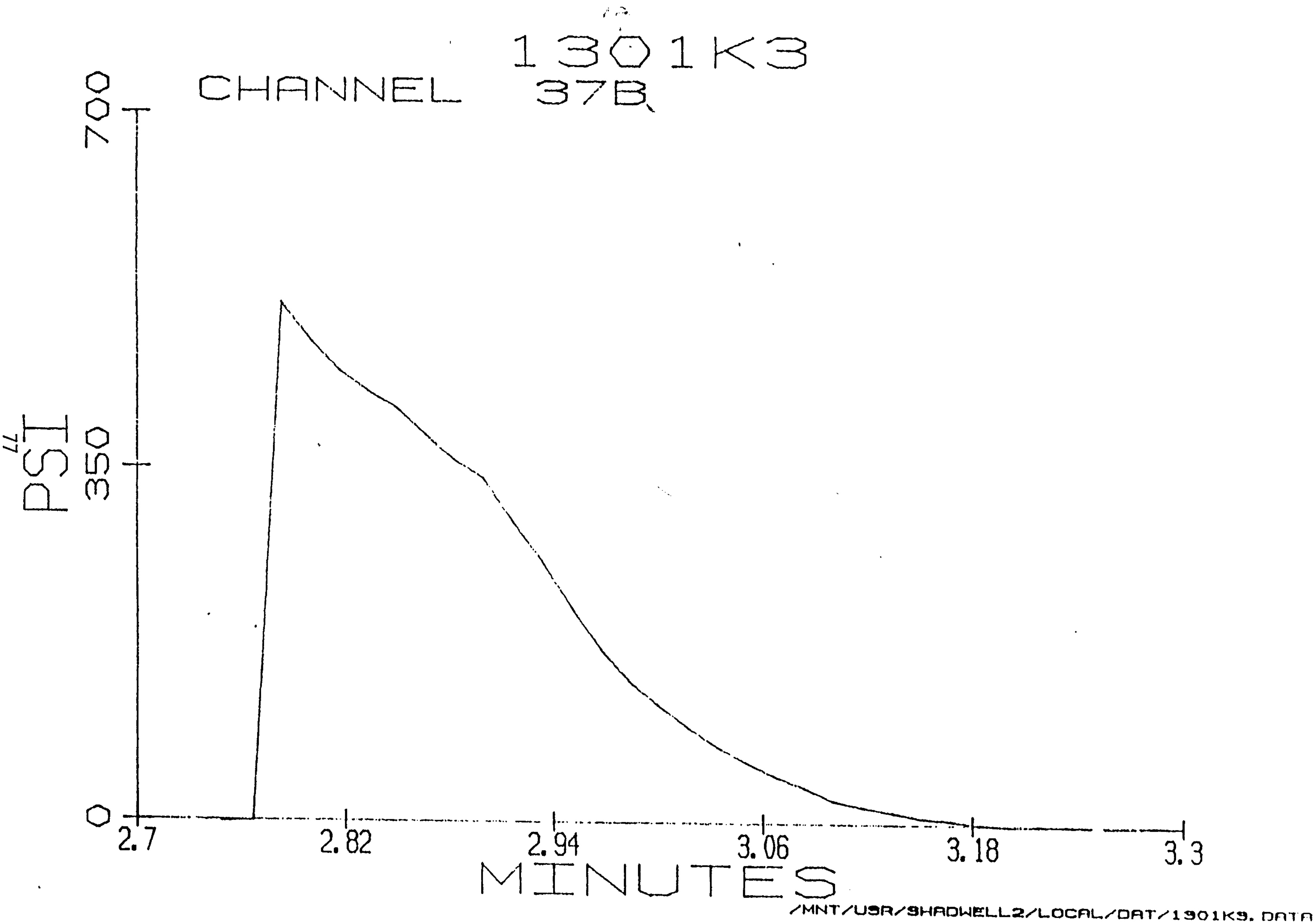
- Target/Use Groups.
- Preliminary Test Data.
- Fire Testing.
- Cold Discharge Testing

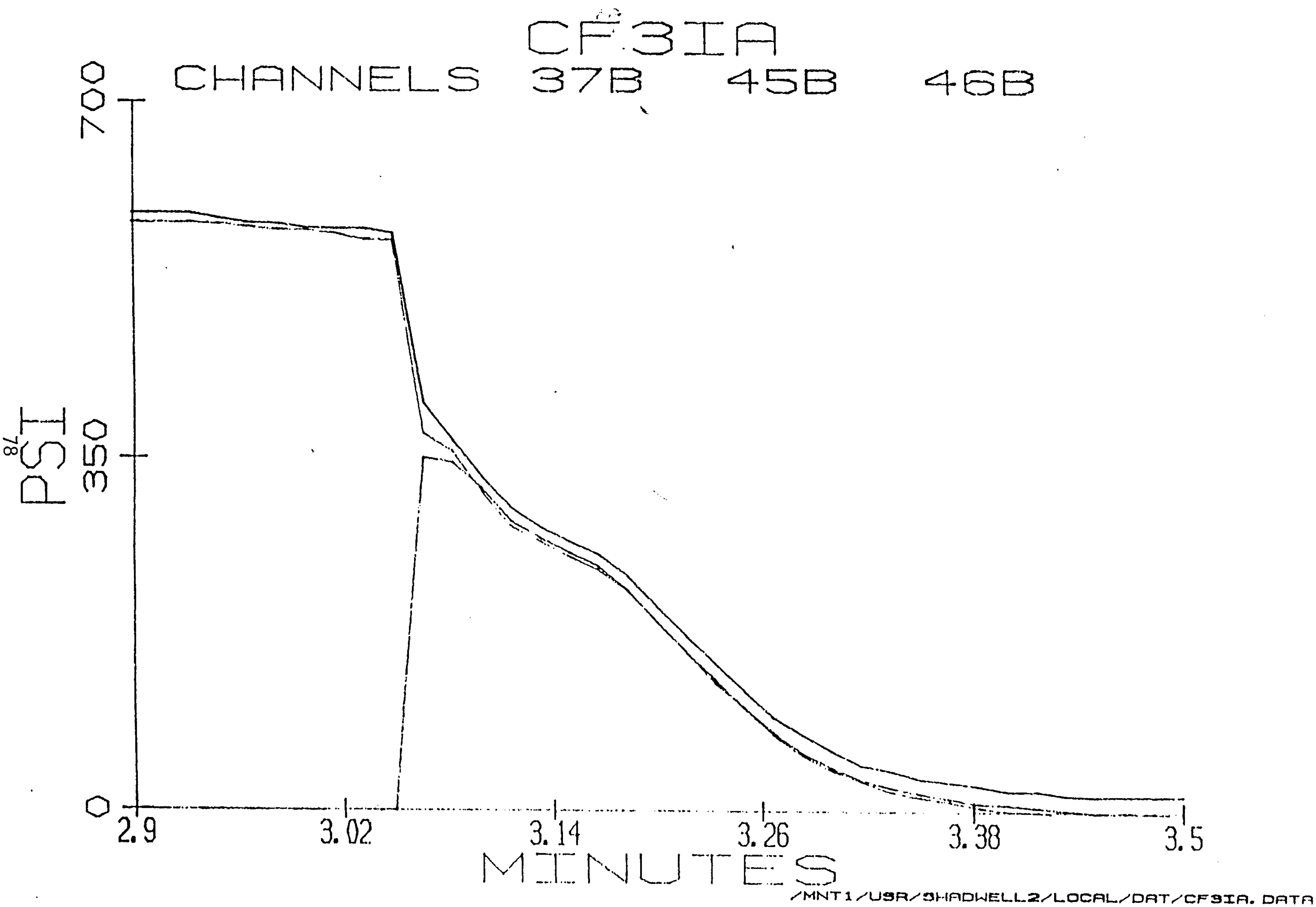
Target/Use Groups for CF_3I

CF_3I Intermediate Scale Testing @ NRL/CBD

- True "Drop In" (North Slope Representatives (NSR))
- Modified Existing Systems
- New Commercial Systems
- DOD Systems

CF$_3$I and Halon 1301 Comparaison NRL/CBD 2000 ft^3 Compartment 2.5 ft^2 N-heptane Pool Fire				
Agent	Design Conc. %	Agent Weight (lbs)	Discharge Time (sec)	Fire Out Time (sec)
Halon 1301	5.09	40.9	4-5	8
CF$_3$I	5.03	53.1	4-5	6

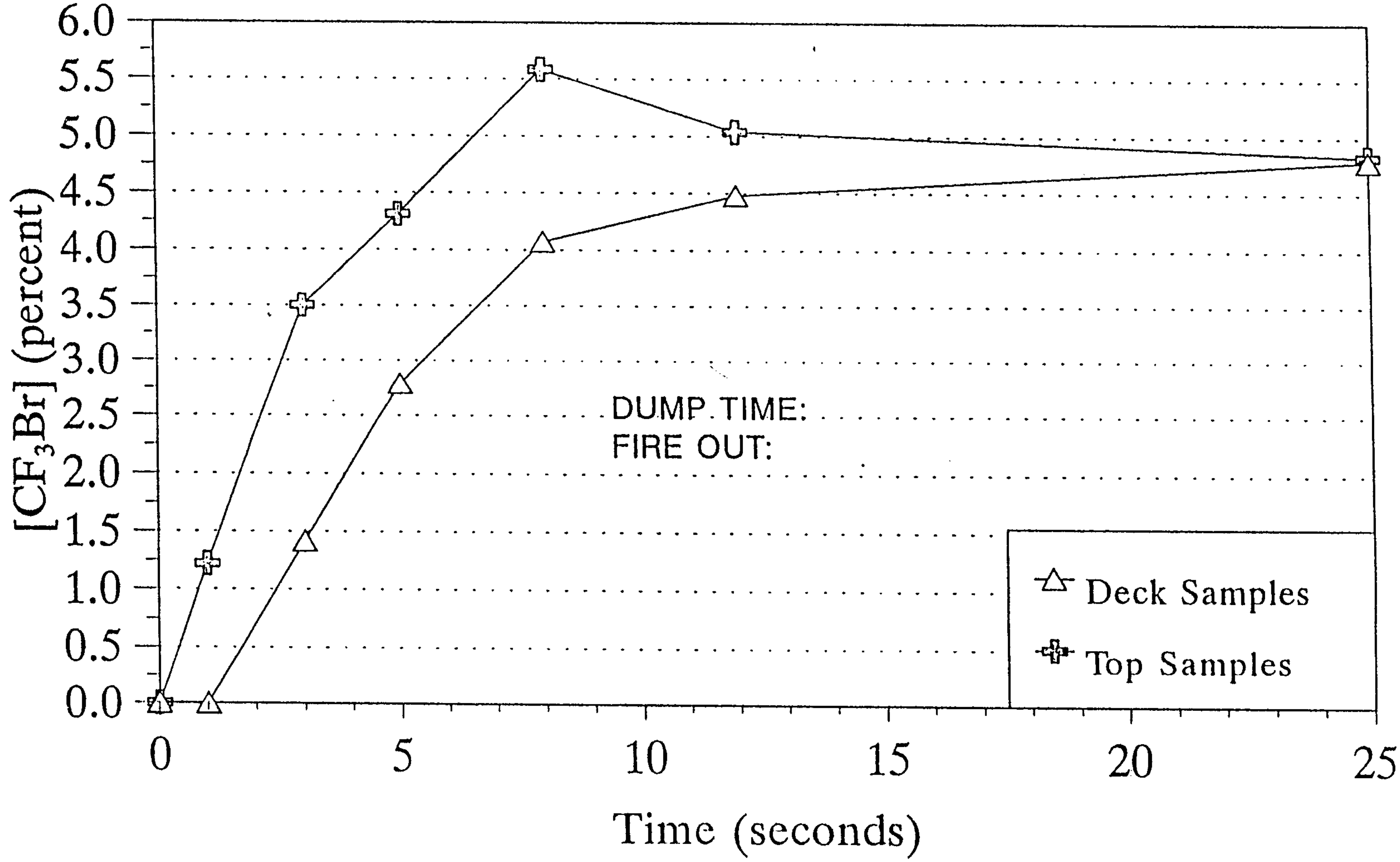

1301K3
CHANNEL 37B
PSI
700
350
0
2.7
2.82
2.94
3.06
3.18
3.3
MINUTES
/MNT/USR/SHADWELL2/LOCAL/DAT/1301K3.DATA


CF3IA
CHANNELS 37B 45B 46B
PSI
700
350
0
2.9
3.02
3.14
3.26
3.38
3.5
MINUTES
/MNT1/USR/SHADWELL2/LOCAL/DAT/CF3IA.DATA

HALON 1301 (CF_3Br) 5.09%

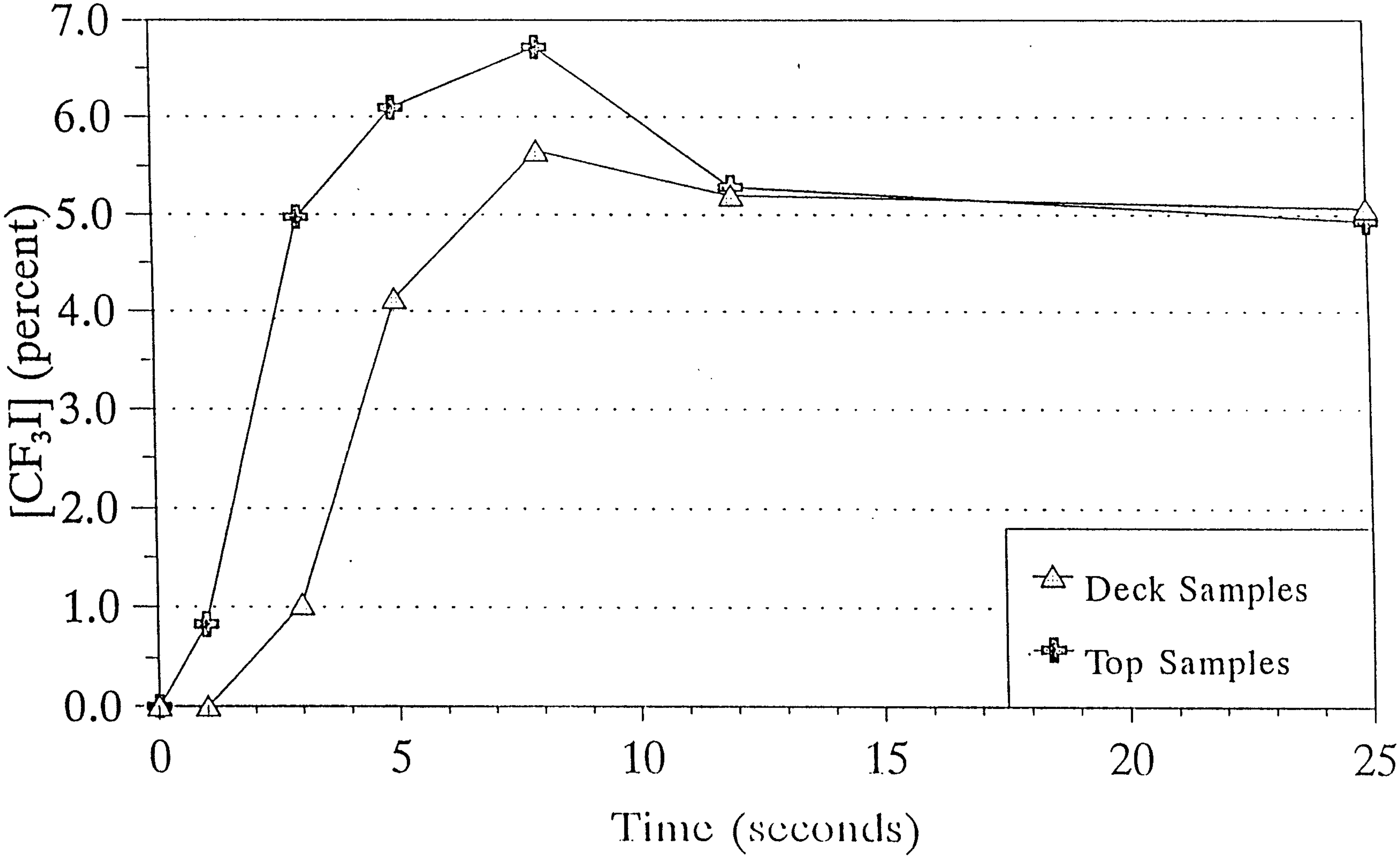
2.5 ft^2 n-Heptane Pan Fire

3/16" Navy Nozzle



1301K3

CF_3I 5.03%
2.5 ft^2 n-Heptane Pan Fire
3/16" Navy Nozzle

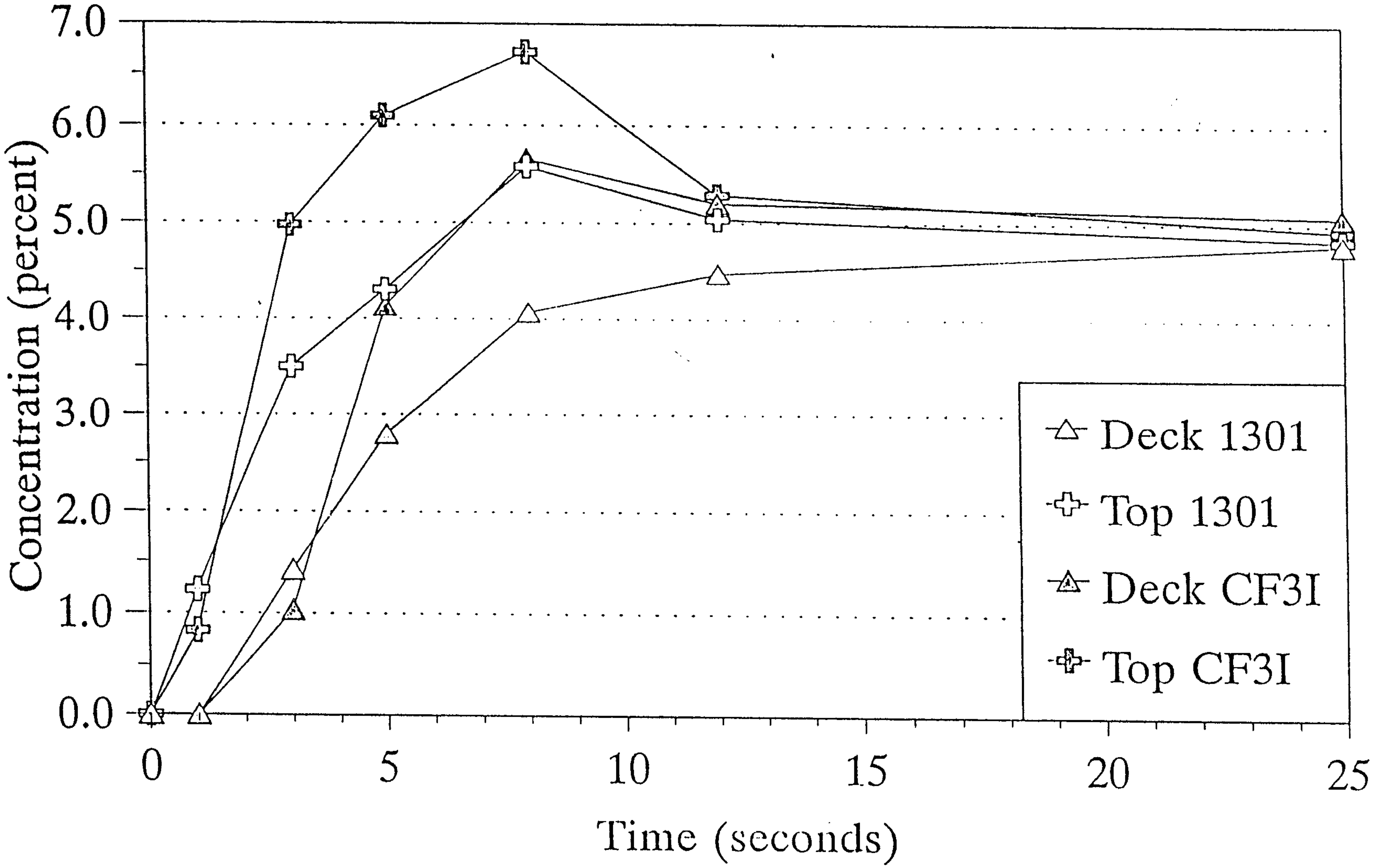


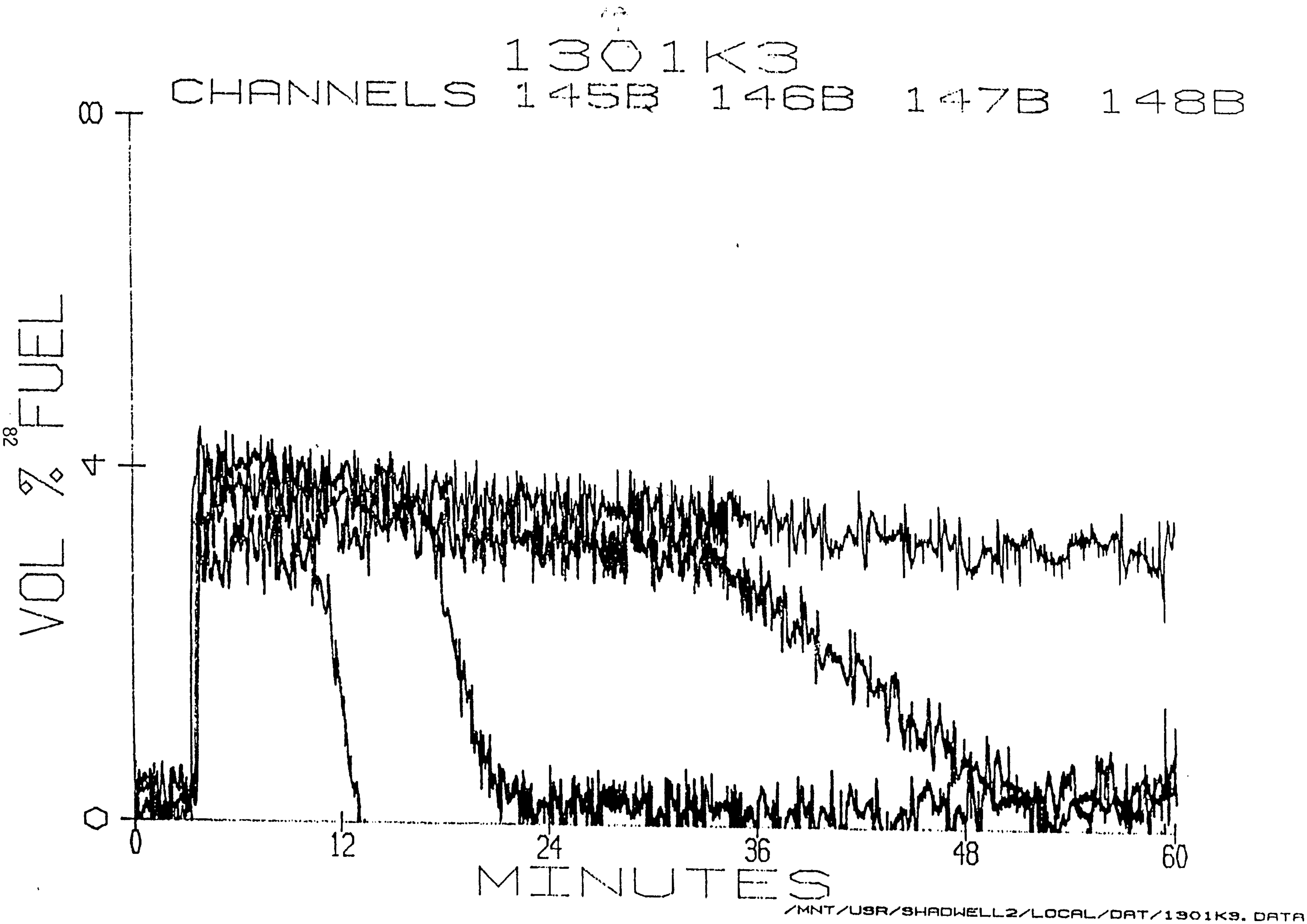
CF3IA

CF_3Br (5.09%) & CF_3I (5.03%)

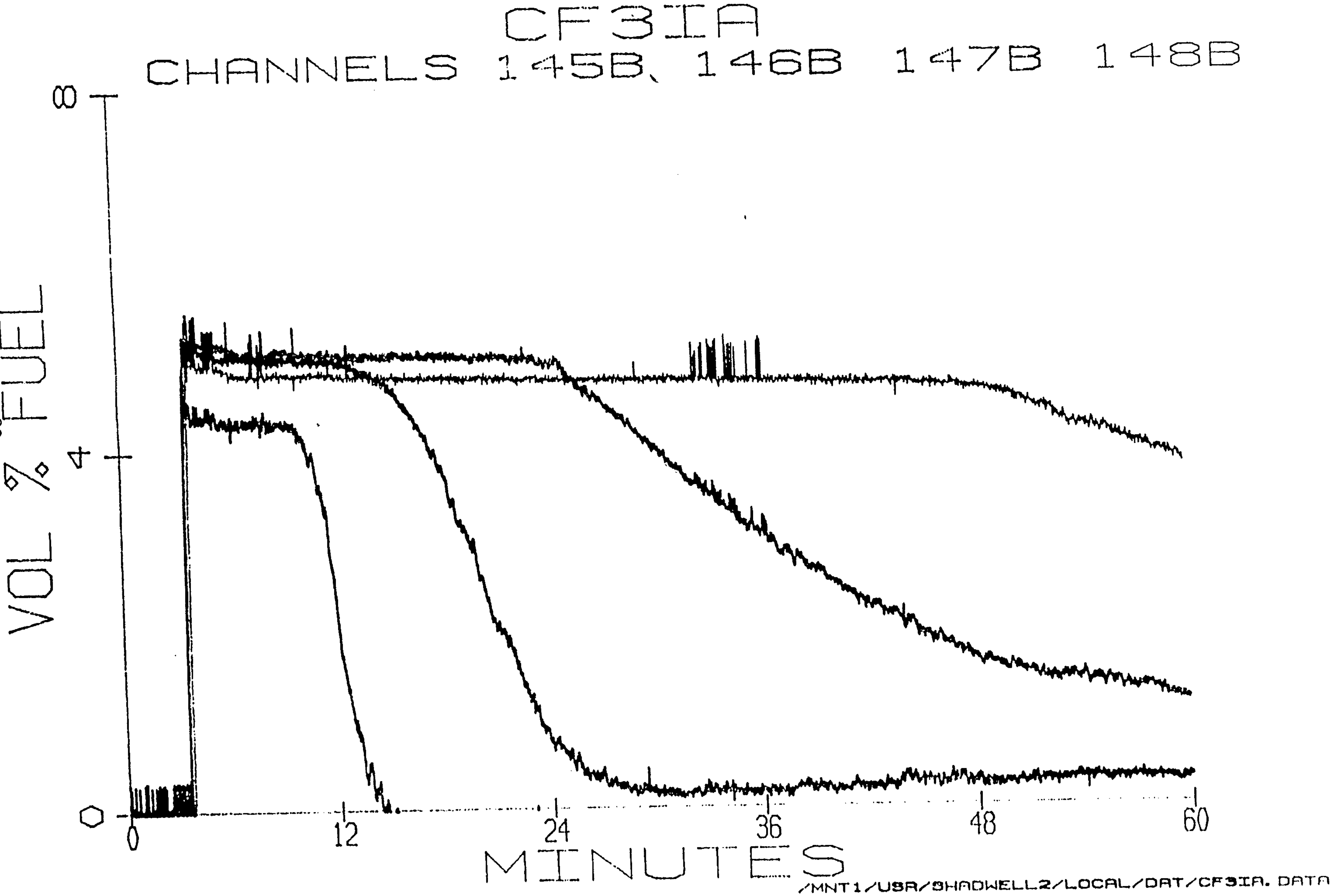
2.5 ft^2 n-Heptane Pan Fire

3/16" Navy Nozzle





1301K3
CHANNELS 145B 146B 147B 148B
VOL % FUEL
8
4
0
0
12
24
36
48
60
MINUTES
/MNT/USR/SHADWELL2/LOCAL/DAT/1301K3. DATA


CF3IA
CHANNELS 145B, 146B 147B 148B
VOL % FUEL
8
4
0
0
12
24
36
48
60
MINUTES
/MNT1/USR/SHADWELL2/LOCAL/DAT/CF3IA.DATA

Fire Testing

CF_3I Intermediate Scale Testing @ NRL/CBD

- 10 Second Baseline Discharge Time.
- Design Concentrations: 4.0 %, 4.5 % , and 5.0 %
- Slow (20 sec) and Fast Discharges (5 sec) @ 4.0 - 5.0 % (exact design concentration to be determined).
- N-heptane 2.5 ft^2 Baseline Pool Fire.
- N-heptane 12 ft^2 Pool Fire with 10 sec Discharge Time @ 4.0 - 5.0 % (exact concentration to be determined).
- 600 PSI Baseline System.
- 300 PSI System.
- Total of 7 Fire Tests.

2000 ft^3 Compartment

Cold Discharge Testing

CF_3I Intermediate Scale Testing @ NRL/CBD

- Baseline 10 Seconds Discharge, 600 PSI, One Nozzle System.
- 300 PSI System.
- Two Nozzle System.
- Short Discharge (3 sec).
- Fast Discharge (20-25 sec).
- SF_6 Comparison.
- Superpressurized (1200 PSI).

12000 ft^3 Compartment

CF_3I NEWSBRIEF No. 5 — 15 September 1994

Air Base Fire Protection and Crash Rescue Section (WL/FIVCF) Editor: Charles Kibert
Tyndall AFB, Florida 32403-5323 Tel: (904) 283-3734 Fax: (904) 283-9797

1. Recent Working Group Meeting

A meeting of the Working Group was held on 23 August 1994 at NMERI at the request of the North Slope companies for the purpose of assessing the impact of recent cardiotoxicity test data and determining a future course of action. The overall agenda of the meeting and a presentation by Stephanie Skaggs are attached to provide a flavor of what was discussed. Lessons learned from the CF_3I development effort were discussed for consideration during future agent development efforts. It was concluded that CF_3I would not meet the needs of many of the members of the group and that there was still a need for an occupied space Halon 1301 replacement that did not require extensive systems modifications.

2. CF_3I Toxicity Data

As is widely known by now, the cardiotoxicity testing for CF_3I produced a LOEL of 0.4% and a NOEL of 0.2%. The result is that CF_3I cannot be used in normally occupied spaces for fire protection. Additionally, genotoxicity testing showed it to be mutagenic in the Ames test and positive in micronucleus testing. A subchronic 90 day test will be required to more clearly define these effects. The details of this testing are being worked out at present.

3. Future Directions

a. Pacific Scientific is continuing to market CF_3I as a Halon 1301 replacement for appropriate applications and a continued supply is assured for the foreseeable future. Many of the critical protection applications are unoccupied spaces and a significant market is still anticipated.

b. The Air Force will test CF_3I as a streaming agent in a series of UL tests about to begin. It will be compared to Halon 1211 and perfluorohexane, along with other leading candidates.

4. New Group Meeting

At the Working Group meeting it was decided to recast the CF_3I Working Group into a new group (Advanced Agent Working Group?) and have the first meeting during the International CFC/Halon Conference in Washington DC. The meeting will take place at the DC Marriott Courtyard, 1900 Connecticut Ave, from 5-7 pm on Sunday 23 October 94. The purpose is to organize to continue the pursuit of an occupied space Halon 1301 replacement on a collaborative basis among users and the research community.

APPENDIX C

DRAFT ASTM PROVISIONAL STANDARD SPECIFICATION FOR TRIFLUOROIODOMETHANE, CF_3I

Provisional Standard Specification for Iodotrifluoromethane, CF_3I[1]

1. Scope

1.1 This provisional standard covers requirements for CF_3I (Iodotrifluoromethane, CAS No. 2314-97-8) as a research and development agent for firefighting and explosion inertion/suppression. This standard is not intended for large scale commercial usage.

1.2 This specification does not address fire fighting equipment or hardware that employs CF_3I or the conditions of employing such equipment (for example, portable extinguishers, fixed fire suppression systems, and explosion inerting systems, etc.).

1.3 This specification does not address the storage or transportation of CF_3I. The storage, handling and transportation issues may be addressed in future ASTM specifications.

[1.4 CF_3I has been found to be a cardiac sensitizer with a NOAEL (no observed adverse effect level) of 0.2%. Therefore, sampling and other procedures should be performed only in areas with effective mechanical ventilation. Long term exposures should be avoided pending the development of appropriate exposure standards.]

1.4[5] <u>This Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.</u>

2. Referenced Documents

2.1 ISO Standards[2]:

ISO3363. Fluorochlorinated, Hydrocarbons for Industrial Use-Determination of Acidity-Titrimetric Method.

---

1 This Standard is under the jurisdiction of the ASTM Committee D-26 on Halogenated Organic Solvents and is the direct responsibility of Sub-Committee D-26.09. On recycled Halon 1301 (Bromotrifluoromethane), and newly manufactured CF_3I (Iodotrifluoromethane).

2 Available from American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

ISO3427. Gaseous Halogenated Hydrocarbons (Liquefied Gases)-Taking of a Sample.

ISO5789. Chlorinated Hydrocarbons for Industrial Use-Determination of Non-Volatile Residue.

2.2 U.S. Military Standards[3]:

Mil-Standard 105 Sampling Procedures and Tables for Inspection by Attributes.

Mil-Standard 129 Marking For Shipment and Storage.

Mil-Standard 1188 Commercial Packaging of Supplies and Equipment.

3. Synonyms

Trifluoroiodomethane, Trifluoromethyl Iodide, FIC-13I1[4], Triodide®[5], Iodoguard ®[6], Halon 13001

4. Material Requirement

4.1 CF_3I shall conform to the requirements prescribed in Table 1 when tested by the appropriate method(s) listed in Section 6.

4.2 By agreement between the purchaser and supplier, analysis may be required and limits established for elements or compounds not specified in the Table 1.

3 Available from Standardization Documents Order Desk, Building 4, Section D, 700 Robbins Avenue, Philadelphia, PA 19111-5904, Attention: N.P.O.B.S.

4 Halocarbon Numbering System

5 Registered Trademark of Pacific Scientific, 1800 Highland Ave., Duarte, CA, 91010.

6 Registered Trademark of West Florida Ordnanace, Inc., 441 N. Jefferson St. Suite 111, Pensacola, FL 35201.

6.2.1 Determine the purity by gas chromatograghy with the technique described in 6.2.2 through 6.2.5 or another acceptable laboratory technique providing equivalent results.

6.2.2 *Apparatus* - One of the following special apparati is required to determine the percent of CF_3I.

6.2.2.1 Gas-Chromatograph, equipped with a 1 mV recorder or comparable data station. The detector should be comprised of a electron ionization (selective ion) detector or thermal conductance detector.

6.2.2.2 Column, 3m x 1/8 in. (3.175mm) outside diameter thin-wall stainless steel tubing, packed with 80 to 100 mesh Porapak Q or equivalent or a column giving equivalent results.[7]

6.2.2.3 Sampling Valve, 5 ml volume.

6.2.3 *Reagents*, The carrier gas shall be a commercial grade of helium. The column packing shall consists of a standard solution, for example, 20% (wt./wt.) practical hexadecane, on 80 to 100 mesh Porapak Q or equivalent.

6.2.4 *Procedure:*

6.2.4.1 Install the column and adjust the temperature of the column oven accordingly. For a gas-chromatogragh with a mass-selective detector set the injection port to 50[°] C, the column temperature to 80[°] C and the detector temperature to 280[°] C. The temperature should be set to rise at 10 C/min with a maximum temperature of 180[°] C. Gas-chromatographs equipped with a thermal conductance detector shall have the column oven set at 80[°] C, the injection port to 160[°] C and the detector block set at 100[°] to 110[°] C. The temperature should be set to rise 10[°] C/min.to a maximum of 180[°] C.

6.2.4.2 The helium flow should be set at 1.0 L/min and 20 L/min for a mass selective detector and thermal conductance detector, respectively.

6.2.4.3 Adjust the thermal conductance detector voltage to 8 V. Allow the instrument to stabilize.

6.2.4.4 Take the sample from the liquid phase (inverted cylinder). Flush the sample loop and sample valve for approximately 2 min before sampling.

---

7 Available from Alltech, 2051 Waukegan Road, Deerfield, IL 60015. Phone 800-255-8324.

6.2.4.5 Rotate the gas sampling valve to transfer the sample into the chromatographic system and note the time.

6.2.4.6 Close the sample cylinder valve.

6.2.4.7 Allow the sample to elute for approximately 18 min, attenuating if necessary to make the peak heights a convenient size (for thermal detector systems). Under proper instrument settings, the sample should elute after approximately 5 min.

6.2.5 *Calculation* - The percent CF_3I shall be calculated as follows:

$$\% CF_3I = \frac{A(CF_3I)}{A_x} \times 100$$

where: $A(CF_3I)$ = area of CF_3I peak, and
A_x = total area of all of the peaks

A CF_3I percent below that specified in Table 1 shall constitute failure of this test.

6.3 *Acidity* - A large sample shall be vaporized in the presence of distilled water. The acidity of the solution shall be determined by the appropriate method specified in ISO 3363, titration given in 6.3.1.2 through 6.3.2.3, a pH indicator, or another acceptable laboratory technique providing equivalent results.

6.3.1 *Sodium Hydroxide Titration:*

6.3.1.1 *Reagents:*

(1) Sodium Hydroxide, 0.01 N solution, standardized against Reagent Grade potassium acid phthalate.

(2) Methyl Red Indicator, 0.1% solution.

6.3.1.2 *Procedure:* Place 50 ml of a crushed ice (made from distilled or deionized water)-distilled or deionized water slurry in a 250-ml stoppered Erlenmeyer flask and add 50 g. of CF_3I to the slurry. Place the stopper in the flask loosely and swirl the flask gently from time to time until the ice is completely melted. Add one drop of methyl red indicator, and if a reddish color remains, titrate to a yellow endpoint with 0.01 N sodium hydroxide solution. Run a crushed ice water blank (with no CF_3I) along with the sample.

6.3.1.3 *Calculation:* The ppm acid ~~acid~~ halides, as HI shall be calculated as follows:

$$\text{ppm acid halides} = \frac{(A - B) \times N_{NaOH} \times 0.0809 \times 10^6}{\text{weight of sample(g.)}}$$

where: N_{NaOH} = Normality of the NaOH solution
A = ml NaOH for sample, and
B = ml NaOH for blank

Acid halides in excess of the amount specified in Table 1 shall constitute failure of this test.

6.3.2 *Acidity by Universal Indicator:*

6.3.2.1 *Apparatus:*

(1) Fritted Glass Sparger, of coarse porosity, contained in a 100 ml glass scrubbing bottle provided with inlet and outlet tubes.

(2) Neoprene Connecting Tubing.

(3) Wet Test Meter. 0.1 ft^3 /revolutions.

(4) Needle Valve Control, No. 55-660, Matheson Co.[8], or equivalent.

6.3.2.2 *Reagent* - Universal Indicator, with color chart, Fisher Scientific Co.[9], or equivalent.

6.3.2.3 *Procedure* - Prepare neutralized distilled water by adding 0.4 ml of Universal Indicator solution to 100 ml of distilled water, and titrate with 0.01 N sodium hydroxide until the water shows a pH of 7.0 when compared to the Universal Color Chart. Add 50 ml of the neutralized water to the glass scrubbing bottle fitted with glass gas sparger. Attach a needle valve control to the CF_3I sample cylinder, connect the cylinder, inverted, to an empty safety trap. Connect the safety trap outlet to the scrubbing bottle inlet. Connect the scrubbing bottle outlet to the wet test meter. Open the needle valve slowly and pass 20 L of sample through the scrubber at a flow rate of approximately 500 ml/min. Turn off the needle valve and disconnect the sample cylinder from the scrubbing bottle. Transfer 10 to 12 ml of the water solution to a clean test tube. Add 0.1 ml of Universal Indicator solution and swirl. Read the pH of the solution by comparison

---

8 Available from Matheson Co., 430-T Cardean Road, Horsham, PA 19044. Phone 215-674-0686.

9 Available from Fisher Scientific Company, Mid-Atlantic Region, 585 A Drive, Pittsburgh, PA 15238. Phone 800-766-7000.

with the Universal Color Chart. Report the pH reading. No observable change in the pH indicates an acidity of less than 1.0 ppm.

6.4 *Water Content* - CF_3I shall be tested for water content. The analysis may be conducted by the phosphorus pentoxide method, infrared absorption, electrolytic moisture analysis, piezoelectric analyzer, high-sensitivity gas analysis tube or other acceptable laboratory technique. The accuracy of the results and the standard method shall be by orthodox Karl Fischer method. Water content greater than that specified in Table 1 shall constitute failure of this test.

6.5 *Test for Halogen Ion (F-and I-)* - A sample shall be tested for the presence of fluoride and iodide ions as specified in 6.5.1 through 6.5.3 or by another acceptable laboratory technique providing equivalent results. Generally, a pH ion-selective electrode shall be used to measure ion content of a neutralized solution of water through which a known amount of CF_3I has been vaporized.

6.5.1 *Apparatus:* The apparatus shall consist of a pH meter capable of measuring millivolts and the appropriate ion selective electrode (fluoride or iodide).

6.5.2 *Reagents:* Fluoride ion (ppm) standard and iodide (ppm) standard.

6.5.3 *Procedure* - The neutralized solution from the acidity determination (6.3) is used for measurement of the fluoride and iodide concentration. Measurements of the CF_3I neutralized solution are to be compared to measurements taken from standard solutions prepared according to manufacturer's directions; diluted to 0.5 ppm ion content for each of the ions, fluoride and iodide respectively. Measurement of fluoride or iodide content greater than the prepared standard shall constitute failure of this test.

6.6 *Suspended Matter and Sediment* - Examine visually for any suspended matter or sediment. Observation of any suspended matter or sediment shall constitute failure of this test.

6.7 *Nonvolatile Residue* - Determine the nonvolatile residue by the method described in ISO 5789 or another acceptable laboratory technique providing equivalent results.

7. Container, Packaging and Package Marking

7.1 Containers used for shipping and storage of CF_3I shall conform to DOT-4BA240.

7.2 Containers used for shipping and storage of CF_3I conforming to this specification shall be marked in accordance with Mil-Std-129, or Mil-Std-1188, whichever is applicable, and with the following information as a minimum.

7.2.1 Suppliers name and address

7.2.2 CF_3I or CF3I

7.2.3 CAS No. 2314-97-8[10]

7.2.3 Statement that the material conforms to PS XXXX.

8. Key Words

8.1 Iodotrifluoromethane; Trifluoromethyl Iodide; CF_3I; FIC-13I1, Firefighting; Firefighting Agent; Fire Protection; Fire Suppressant; Iodofluoronated Hydrocarbon; Fluoroiodocarbon; CAS No. 2314-97-8.

10 Chemical Abstracts Service Registry Number